# Teck



## Fugitive Dust Risk Management Plan

## 2021 Annual Report

Red Dog Operations Teck Alaska Incorporated December 2022

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## **Acronyms and Abbreviations**

| CAKR     | Cape Krusenstern National Monument              |
|----------|---|
| CSB      | Concentrate Storage Building                    |
| DEC      | Alaska Department of Environmental Conservation |
| DMTS     | DeLong Mountain Transportation System           |
| DNR      | Alaska Department of Natural Resources          |
| MP       | mile post                                       |
| NANA     | NANA Regional Corporation                       |
| NPS      | National Park Service                           |
| OPA      | Onion Portage Adventures                        |
| PAC      | Personnel Accommodations Complex                |
| PMC      | Plant Materials Center                          |
| RDO      | Red Dog Operations                              |
| RDTWG    | Red Dog Tundra Working Group                    |
| RMP      | Fugitive Dust Risk Management Plan              |
| T-Dam    | Main Tailings Dam                               |
| TEOM     | tapered element oscillating microbalance        |
| Terramac | Terramac 9T Crawler                             |
| TSP      | total suspended particulates                    |
| TUB      | Truck Unloading Building                        |
| Tuuq     | Tuuq Drilling, LLC                              |
| VEE      | visible emissions evaluation                    |
| XRF      | x-ray fluorescence analyzer                     |



## Summary

This document presents the 2021 Fugitive Dust Risk Management Plan (RMP) Annual Report for Red Dog Operations (RDO), including the mine, and road and port areas that make up the DeLong Mountain Transportation System (DMTS). The goal of the RDO Fugitive Dust Risk Management Program [overarching goal: *Minimize risk to human health and the environment surrounding the DMTS and outside the Red Dog Mine boundary over the life of the mine*] is to ensure that dust levels remain low using the practices discussed in this 2021 report. This report presents results from efforts related to each of the risk management implementation plans, including the Communication Plan, Dust Emissions Reduction Plan, Remediation Plan, Worker Dust Protection Plan, Uncertainty Reduction Plan, and Monitoring Plan. Activities are summarized below in relation to each of these plans.

The Communication Plan centers around maintaining clear communication with local communities and other interested parties about fugitive dust risk management efforts at the mine. Communication Plan activities during 2021 included community meetings, Subsistence Committee meetings, and communications with other stakeholders and organizations who expressed an interest in mine operations. Details are presented in the section titled "Communication Actions," below.

The Dust Emissions Reduction Plan in 2021 included application of calcium chloride and water on roads to reduce dust, use of the "waterless" air truck wash at the port site, a truck wash located at the mine site in summer months, tailings beach dust suppression, and installation of a new water source for the mine water truck. Details are presented in the section titled "Dust Emission Reduction Actions," below.

Activities related to the Remediation and Reclamation Plan in 2021 involved revisiting previously remediated sites to determine if recovery was progressing in accordance with Alaska Department of Environmental Conservation (DEC) Spill Prevention and Response goals. The Red Dog Tundra Working Group (RDTWG) focuses on rehabilitating spill sites along the DMTS; in 2021, the group met at Red Dog for a site tour and to discuss reclamation studies. Native seed harvest was completed along the DMTS. Details are presented in the section titled "Remediation Actions," below.

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Activities related to the Worker Dust Protection Plan include ongoing programs designed to monitor and minimize workers' exposure to dust while at Red Dog, and to facilitate comprehensive communication about these programs, policies, and practices. In 2021, worker health monitoring continued through regular blood lead level testing and by environmental monitoring performed by the on-site Safety & Health department. Strictly enforced policies remain in place to ensure that worker health is protected and that all work environments are safe. Teck Alaska, Inc. (Teck) takes employee health extremely seriously, and noncompliance with health and safety policies is not tolerated. Details are presented in the section titled "Worker Dust Protection Actions," below.

Activities related to the Uncertainty Reduction Plan include research or studies to reduce uncertainties related to the assessment and management of risk to humans and the environment. In 2020, a white paper was prepared to update the state of knowledge based on soil bioaccessibility testing at RDO. The draft paper was provided as an appendix to the 2020 Annual Report. Comments were received back and responses are included in this report.

Activities related to the Monitoring Plan are intended to provide the necessary operational and environmental monitoring data to facilitate continued reduction of fugitive metals and dust emissions, verify the continued safety of caribou and other subsistence foods and water, as well as the health of ecological environments and habitats in the vicinity of the mine, road, and port. In 2021, monitoring activities proceeded on schedule, and statistical analyses were performed on multi-year data sets to identify and evaluate trends and patterns. In 2021, the following monitoring programs were implemented:

- Visual emissions evaluations
- Source monitoring at the mine and port with real-time air samplers
- Real-time alarm system monitoring for dust at the mine
- Road surface monitoring to assess tracking of metals
- Dustfall jar monitoring at the mine, road, and port

Along the DMTS road, trends in concentrations of lead and zinc concentrations have either decreased significantly or show no change. At the mine and port site, trends in lead and zinc concentrations have significantly increased over the past four-year period. The 2021 results indicate some increases noted

over the last four-year period, indicating that additional attention to potential dust controls will need to be considered in 2022.



## Introduction

In accordance with the Fugitive Dust RMP (Exponent 2008), the purpose of this report is to provide a summary of risk management activities conducted at the Red Dog operation in the prior calendar year.

## Background

The Red Dog Mine is approximately 50 miles inland of the Chukchi Sea, in the western end of the Brooks Range of Northern Alaska. The mine is located on land owned by the NANA Regional Corporation (NANA) and operated by Teck. Base metal mineralization occurs naturally throughout much of the western Brooks Range, and elevated zinc, lead and silver concentrations have been identified in many areas (Exponent 2007). The Red Dog Mine has been in operation since December 1989.

At the mine, ore containing lead and zinc sulfides is mined and milled to produce lead and zinc concentrates in a powder form. These concentrates are hauled year-round from the mine via the DMTS road to Concentrate Storage Buildings (CSBs) at the port, where they are stored until being loaded onto ships during the summer months when the port is ice-free. The storage capacity allows mine operations to continue year-round. During the shipping season, the concentrates from the storage buildings are loaded into an enclosed conveyor system and transferred to the shiploader, and then into barges. The barges have built-in and enclosed conveyors that are used to transfer the concentrates to the holds of deepwater ships. The DMTS road passes through the Cape Krusenstern National Monument (CAKR), which is managed by the National Park Service (NPS). A study conducted by NPS in 2000 found elevated levels of metals in moss near the DMTS road, declining with distance from the road (Ford and Hasselbach 2001).

In response to the results from the NPS study, Teck conducted studies to characterize the dust issue throughout the mine, road, and port areas, and subsequently conducted a human health and ecological risk assessment (Exponent 2007) to estimate possible risks to human and

ecological receptors<sup>1</sup> posed by exposure to metals in soil, water, sediments, and plants and animals in areas surrounding the DMTS, and in areas surrounding the Red Dog Mine ambient air/solid waste permit boundary and port site. The human health risk assessment evaluated potential exposure to DMTS-related metals through incidental soil ingestion, water ingestion, and subsistence food consumption under three scenarios: 1) child subsistence use, 2) adult subsistence use, and 3) combined worker/subsistence use.

The human health risk assessment, including the subsistence foods evaluations, found that it was safe to continue harvesting of subsistence foods from all areas surrounding the DMTS and mine, including in unrestricted areas near the DMTS, without restrictions. Although harvesting remains off limits within the DMTS, human health risks were not elevated even when data from restricted areas were included in the risk estimates.

The ecological risk assessment evaluated potential risks to ecological receptors inhabiting terrestrial, freshwater stream and pond, coastal lagoon, and marine environments from exposure to DMTS-related metals. The ecological risk assessment found that:

- In the tundra environment, changes in plant community composition (for example, decreased lichen cover) were observed near the road, port, and mine, although it was not clear to what extent those effects may have resulted from metals in fugitive dust, or from other chemical and physical effects typical of dust from gravel roads in Alaska.
- The likelihood of risk to populations of animals was considered low, with the exception of possible risks related to lead exposure for ptarmigan living closest to the port and mine.
- No harmful effects were observed or predicted in the marine, coastal lagoon, freshwater stream, and tundra pond environments, although the potential for effects to invertebrates and plants could not be ruled out for some small, shallow ponds found close to facilities within the port site. However, no evidence of adverse effects



<sup>&</sup>lt;sup>1</sup> Plants and animals

was observed in these port site ponds during field sampling conducted as part of the risk assessment.

Subsequent to completion of the risk assessment, Teck prepared an (RMP) designed to minimize the potential for effects on human health and the environment over the remaining mine life and beyond (Exponent 2008).

## Risk Management Plan Overview

Based on the results of the risk assessment and stakeholder input on risk management objectives, the RMP was developed to combine and build upon prior and ongoing efforts by Teck to reduce dust emissions and minimize potential effects to human health and the environment over the life of the mine. Specifically, the overarching risk management goal is to: *Minimize risk to human health and the environment surrounding the DMTS and outside the Red Dog Mine boundary over the life of the mine.*<sup>2</sup>

Although human health risks were not found to be elevated, and potential ecological risks were found to be limited, conditions may change over time, and this possibility was also considered in the design of the RMP. Future changes in conditions and in potential human and ecological exposures over the life of the operation can be addressed through implementation of risk management, dust emissions control, and monitoring activities. More specifically, the RMP established a set of seven risk management objectives (Exponent 2008), which formed the basis for preparation of six implementation plans. Each of the six implementation plans addresses one or several of the overall objectives of the RMP (Figure 1) and includes the planned scope of work to achieve the objectives.

This annual report assumes that the reader has some familiarity with the Fugitive Dust Risk Management Program and is therefore not intended to be a thorough discussion of that program, nor is it intended to provide complete background on either the risk management program or risk assessment that led to the development of the RMP. To develop a more

<sup>&</sup>lt;sup>2</sup> The mine closure and reclamation plan addresses risk management within the mine solid waste permit boundary (collocated with the ambient air boundary, see Figure 3).

thorough understanding of the risk management programs, interested parties are encouraged to review the human health and ecological risk assessment documents (Exponent 2007), as well as the RMP (Exponent 2008) and its component implementation plans:

- Communication Plan (Exponent 2010)
- Dust Emissions Reduction Plan (Exponent 2011a)
- Remediation Plan (Exponent 2011b)
- Worker Dust Protection Plan (Exponent 2011c)
- Monitoring Plan (Exponent 2014)
- Uncertainty Reduction Plan (Exponent 2012)

These plans are available for review under "News and Related Documents" at <a href="http://www.teck.com/operations/united-states/operations/red-dog/">http://www.teck.com/operations/united-states/operations/red-dog/</a>.

## Data Collection and Reporting Objectives

The risk management program includes collection of large amounts of data for the various implementation plans (discussed below) that are intended for either operational or regulatory purposes. Data collected for operational purposes are intended to provide Teck with information on the effectiveness of dust emissions control and reduction efforts. Data collected for regulatory purposes are intended to provide Alaska DEC with the necessary information to verify that conditions are protective of human health and the environment.

The soil monitoring and marine sediment monitoring programs (described in the section below regarding the summary of monitoring results) are intended to satisfy a number of requirements, including the regulatory requirements under DEC Contaminated Sites Program, pursuant to 18 AAC 75.360. These two monitoring programs are intended to provide DEC with a means to continue oversight and implement enforcement actions as needed. As such, the results of these programs are formally documented in separate reports to DEC after each monitoring event. Sediment monitoring occurs once every two years, and soil monitoring occurs once every three years. The next sediment sampling event is scheduled for 2022, and the next soil sampling event is scheduled for 2023.



## **Report Organization**

The annual report summarizes work that was conducted during the 2021 calendar year related to each of the implementation plans that are part of the overall RMP. Sections that follow document the communication, dust emissions reduction, remediation, worker dust protection, uncertainty reduction, and monitoring actions taken in 2021.



## **Risk Management Actions Taken in 2021**

The following sections of this annual report summarize each implementation plan, the corresponding risk management objectives, and the actions taken during the 2021 calendar year toward achieving these objectives.

## **Communication Actions**

The Communication Plan follows from RMP Objective #6: *Improve collaboration and communication among all stakeholders to increase the level of awareness and understanding of fugitive dust issues*. To achieve this objective, the Communication Plan was developed with the goal: "To establish consistent methods for communication and collaboration among stakeholders regarding efforts related to dust emission issues." The plan identified multiple types of communication actions, within three categories: communication, collaboration, and education and outreach. A number of methods from these three categories have been implemented as part of the various risk management programs within the RMP.

The following sections outline actions that were taken in 2021 by the Red Dog Environmental and Communication Relations Department to increase communication and participation between RDO and the communities, and to ensure that information is being communicated to all stakeholders and interested parties in an effective manner:

## **Community Meetings**

Community meetings typically provide the opportunity for Red Dog to give the communities updated information on operations, to learn from attendees, discuss concerns, and to discover what questions community members have about Red Dog. In 2021, these meetings were conducted virtually. Below are examples of meetings and events that occurred in 2021:

- Exploration engagement meetings with the Kivalina IRA, Noatak IRA, and Red Dog Subsistence Committee
- Joint Leadership meetings with the Kivalina IRA, Kivalina City Council, and Noatak IRA Council



- Water discharge management engagement meetings with Kivalina IRA, Kivalina City Council, and Noatak IRA Council
- Multiple meetings with the Siñġagmiut Working Group (The Working Group was formed to address environmental concerns, human health issues, traditional land use, and other topics decided on by the Kivalina representatives. To date, topics have focused on water quality testing in the community, tailings dam information sharing, human health studies and employment.)
- Eight helicopter overflights to monitor pre-discharge and post-discharge conditions for Kivalina residents
- Nine Village Fuel Program fuel transfers with the community of Noatak
- 2021 Kivalina Whaling Captains Gas Donation (where 10 whaling captains received two drums of gas each)
- 2021 Marine Mammal Hunter Gas Donation (where 11 Noatak hunters received two drums of gas each)
- Five meetings with Selawik and DEC in response to Selawik Oil Spill
- Eight meetings with the Northwest Arctic Borough School District Youth Leaders program
- Two Red Dog Community Engagement meetings with NANA
- Village Improvement Commission meetings
- Northwest Arctic Borough and Assembly Meetings
- One Annual Hunters meeting in Kivalina
- Nine Village Fuel Program fuel transfers with the community of Noatak
- Four NANA and Teck Community Engagement meetings
- Six Community Wellness Taskforce work sessions where updates were delivered on regional wellness progress at Regional Elders Council meetings
- Caribou Hunter Success Working Group Meeting



• Forty-two contributions for a total of \$702,777 for the year, including \$19,930 in-kind contributions and \$150,000 dedicated specifically to COVID-19 relief funds.

Examples of contributions are as follows:

- Access to food
- Heating fuel and utilities
- Quarantine and testing expenses
- Capacity support for testing locations
- Kobuk 440 supporting wellness through Inupiaq tradition
- Emergency safe drinking water—Ambler
- Emergency fuel spill response—Selawik
- Emergency response to flooding—Buckland
- Community funeral support
- Community spring cleanup
- Community 4<sup>th</sup> of July activities
- Regional back to school activities
- Noatak Student Activities Fund
- Iron Dog Hospitality House
- USMC Toys for Tots at Christmas
- Thanksgiving and Christmas community events
- Noatak Hasty Crew emergency response equipment
- Aqqaluk Trust Youth Leadership Development Social Media Outreach

#### Subsistence Committee Meetings

The Red Dog/NANA Subsistence Committee is an advisory committee made up of hunters and Elders from Noatak and Kivalina. The committee shares traditional knowledge with Red Dog Mine operators and discusses possible effects of mine operations on subsistence activities. Red Dog holds quarterly meetings with the Subsistence Committee. This provides a key opportunity to obtain input from traditional ecological knowledge holders and Elders.

In 2021, discussions were centered around the following:

 Past Spill Site Reclamation—Reclamation at historical spill sites (previously cleaned up to DEC Standards) was discussed with the Subsistence Committee. A review of the RDTWG meeting that occurred in July 2021 was provided. RDO has voluntarily updated the Tundra Treatment Guidelines document and presented the tracked changes to the DEC, to reflect how climate change may be changing best management practices for reclamation in the Northwest Arctic.



- Port CSB Building Roof Residing—Panels on the CSB #1 roof and walls at the port are going to be replaced. The Subsistence Committee previously selected gray as the color. All old metal panels were washed, and sent offsite to a scrap metal recycler.
- Marine Mammal Harvest—RDO voluntarily delays all shipping season activities and vessels from approaching the port in early spring until the Subsistence Committee meets with RDO and gives permission to bring in vessels for the summer port activities, including the shipping. The goal is to minimize potential impacts to the local community's subsistence hunting activities for marine mammals, including beluga, walrus, and Ugruk (bearded seal). Multiple meetings were conducted leading up to the start of the shipping season to ensure that all parties were communicating about the location and status of beluga, walrus, and Ugruk hunting activities. In 2021, the voluntary delay was the longest ever recorded in RDO history, and the Subsistence Committee mentioned its appreciation for the care RDO provided with regard to subsistence hunting.

## **Dust Emissions Reduction Actions**

The Dust Emissions Reduction Plan is intended to achieve RMP Objective #1: *Continue reducing fugitive metals emissions and dust emissions*. In order to achieve this objective, the Dust Emissions Reduction Plan was developed with the goal: "To reduce the amount of fugitive dust released into the environment near the DMTS and Red Dog Mine to protect human health and the environment."

#### **Road Dust Emissions Reduction Actions**

During the warmer months when snow and ice are no longer present, calcium chloride is applied to the gravel roads as a dust suppressant because it retains moisture for prolonged periods. In addition, water trucks spread water on the port and mine site roads. Using the calcium chloride with water applications holds down dust and stabilizes unpaved road surfaces.

#### Tailings Beach Dust Suppression

In 2021, an ice cap was built over the tailings beach for dust suppression for the first time, to trial ice as a new method of dust suppression. Tuuq Drilling, LLC (Tuuq) was contracted to

complete dust suppression application on the Tailings Beaches. Tuuq applied 74,000 gallons of water to the tailings beach surface using a Terramac 9T Crawler (Terramac) from October 18 to October 21, 2021. The Terramac has a 2,000-gallon tank, which was filled using a water truck. The 3-inch pump, located at the rear, is attached to a sprayer nozzle that is used to apply water for creating an ice cap on the tailings. The sprayer nozzle creates a ~25-ft fan of water directly behind the Terramac. The Terramac continued to apply water in this manner until it was 100 ft away from open water/ice or until the tailings were deemed too saturated to safely traverse. To safely apply water within the non-trafficable area, the nozzle was directed 90 degrees to the Terramac's travel direction during the last pass on the tailings beach.

Dust suppression application occurred on the Wing Wall Tailings Beach on October 22, 2021, in a different manner, because the tailings were too soft and therefore the Terramac was unable to travel directly on the tailings. Therefore, Tuuq applied 36,000 gallons of water to the tailings surface using a Terramac and a sprinkler system. The 3-inch pump on the Terramac was attached to a sprinkler to apply water from the wing wall crest. The sprinkler created a ~50-ft fan of water in a semi-circle from the upstream crest. Once an area was sufficiently covered with water, the sprinkler was manually moved along the crest and the Terramac would drive to the next location. This method was utilized because the tailings in that area were too saturated for the Terramac to safely travel.





#### Year-Round Air Wash

In 2021, the "waterless" truck wash at the port Truck Unloading Building (TUB) continued to be used. The air wash uses high-powered blowers to remove residual dust from the trucks following loading and before exiting the TUB. The system designed for the TUB consists of six high-powered air blowers that are typically used to dry cars in automatic car washes.

## Mine Area Dust Emissions Reduction Projects

A dust suppression product trial for HAULAGE-DC<sup>™</sup> was considered for 2021, but safety concerns (potential truck slippage) put the trial on hold.

## **Remediation Actions**

The Remediation Plan is intended to facilitate the achievement of RMP Objective #2: *Continue remediation or reclamation of selected areas to reduce human and ecological exposure.* To achieve this objective, the remediation plan was developed with the goal: "To define a consistent method for identifying and selecting affected areas and implementing remediation and/or reclamation." Specific requirements for remediation are set forth in various permits and approved documents such as the Red Dog Reclamation and Closure Plan.

## Harvesting Seeds for Red Dog Reclamation Projects

Since 2015, Teck and NANA have invested in training, seed harvests, and seed testing, to support development of local native seed market in the NANA Region to sell seed to Teck to revegetate disturbed lands at the mine. Through 2019, seed harvest training was offered in Noatak, resulting in a group of trained harvesters who consistently produced high quality pure, live seed from several local native forbs (herbaceous flowering plants). The COVID pandemic prohibited supervised harvests in Noatak in 2020 and 2021, and despite multi-year efforts by Teck to support seed harvest, no independent harvests have been conducted to date.

As part of RDO's biodiversity management plan, and best reclamation practices, locally adapted native seed is highly desirable to maintain biodiversity in reclamation projects, and to yield successful revegetation of disturbed lands. Seeds from herbaceous flowering plants (referred to as forbs) are a valuable component of reclamation projects. Locally adapted forbs, specifically



those grown near sites that require reclamation, are preferred. Described below is a synopsis of multiple efforts made by Teck in 2021 to acquire seeds for upcoming reclamation projects along the road, and at material sites.

#### Noatak Seed Harvest Program

Teck and NANA began the Noatak Seed Harvest Program in 2015 to procure locally sourced native species to revegetate areas slated for reclamation at Red Dog Mine. In 2021, due to COVID-19, travel to Noatak was restricted or prohibited and therefore no in-person outreach was conducted; instead, seed procurement outreach activities were limited to the following:

- Phone calls to previously trained harvesters
- The Noatak Announcements Facebook Group
- Posting of paper posters in the community.

Despite these efforts, no seed was collected in Noatak in 2021.

#### Red Dog Seed Harvest Program

Due to COVID-19, the Noatak Seed Harvest Program did not occur in 2020 or 2021. Instead, 2021 seed harvest was instead focused at RDO along the DMTS road, as a continuation of 2020 activities. In 2021, NANA-owned and State-owned lands were evaluated for potential harvest species. Based on 2020 observations, it was inferred that mid-July would yield a higher abundance of seed than in the prior year. Unfortunately, in 2021, heavy rains and winds may have contributed to limited availability of seed as most seed had already cast. Tussock cottongrass (*Eriophorum vaginatum*) was harvested from the area surrounding the Gas Exploration sites, and frigid sweet coltsfoot (*Petasites frigidus*) was harvested in the vicinity of DMTS Mile Post 3. The Alaska Plant Materials Center (PMC) cleaned and tested all seed harvested in 2021, to yield a total of 20.7 grams of pure live *E. vaginatum* seed and 2.1 grams of pure live *P. frigidus* seed. The goal was to identify and harvest seed to revegetate spill sites along the DMTS.



#### Kivalina Native Seed Reconnaissance Study

Past conversations with Kivalina residents indicated there might be interest in creating a Kivalina Seed Collection Program, similar to what was developed in Noatak. Teck was interested in knowing if conveniently located potential seed harvest areas were present in the vicinity of Kivalina. Teck hired f&t in July 2021 to do some visual observations and reconnaissance along the lower Wulik River, at the K-Hill material site, and along the evacuation route to the new school site. Lands along the evacuation route consisted largely of wetlands and ponds, and potential cottongrass harvest areas were noted. Additional opportunistic observations were made in August 2021 at the K-Hill material site, and more reconnaissance is planned for 2022.

#### Ambler Native Seed Reconnaissance Study

In 2021, Onion Portage Adventures (OPA), a local and licensed business that is currently developing a berry harvest and sales business in Ambler, Alaska, expressed interest in selling seed to Teck. Teck supported a seed reconnaissance study in Ambler in 2021 to identify potential harvest species and train OPA to harvest seed. OPA provided local transportation, room and board, and a field crew to support the Study. The study was conducted August 9–11, 2022. The goals of the study were as follows:

- Train OPA to identify forbs and grasses suitable for revegetation at Red Dog Mine ("target species") and evaluate seed maturity.
- Identify populations of target species available in harvest quantities.
- Document the growth phase (phenology) of one or more non-target flowering plants.
- Provide OPA with information to determine if seed harvest is viable and consistent with its business model.
- Potentially harvest one or more target species to evaluate purity, germination, and yield seed for revegetation at Red Dog Mine.

To ensure seeds were harvested from plants in a sustainable manner, no more than one of every three stems was harvested in each stand, or approximately 30% of the total seed heads

present. Kituq Williams and Breanna Sheldon of OPA were a vital part of the field crew and provided subsistence knowledge and, if known, Inupiaq names for local plants at the allotments. In addition, Helena Jones and Lula Sheldon, two Inupiat elders from the Ambler community, were interviewed for their traditional knowledge on potential subsistence uses of the target plants identified in the field.

The PMC cleaned the seed and performed purity and standard germination tests on each species ("AOSA Rules for Testing Seeds," Method 20-30C (Association of Official Seed Analysts, 2016). A few species were identified that OPA could harvest and, after proper cleaning and testing at the Alaska PMC, sell to Teck.

The study was conducted on a total of 11 Native allotments in the village of Ambler and on the Ambler and Kobuk rivers to identify native forbs and locate potential harvest sites. One of the land allotments has already been cleared and leveled, and if non-native species are fully eradicated, the plot may provide suitable farming conditions for local seed. No NANA lands were accessed, crossed, or evaluated during this study.

#### Native Seed Harvest Booklets

Seed harvesting in the region will continue to be a critical component of reclamation success at RDO. Collection of local native forbs and grasses that are adapted to local habitat will reduce the potential introduction of non-native invasive plant species potentially found in commercial seed supplies. To assist with future plant collections, three separate field guides with photographs and information on desirable reclamation plants in Ambler, in Noatak, and at RDO. The pocket booklets were created in 2021 and also include information on traditional uses, traditional names, and photographs of each species.

#### **Non-Resident Plant Species**

In summer 2020, RDO removed commercial grass cultivars that were previously used to revegetate historical spill sites (MP-13.2 and MP-35.4). The grasses had created a thick layer of plant material that seemed to prevent colonization of the area by other tundra plants. The cleared areas were backfilled in 2020. In summer 2021, RDO conducted visual evaluations at the following mile posts along the DMTS: MP-3.1, MP-10.5, MP-13.2, MP-26, MP-27, MP-28.2,

MP-34, MP-35.4, MP-48.5, and MP-48.6. The evaluation consisted of visually observing the excavated areas within spill site boundaries, and within an approximately 30-m radius from the excavation limit into the undisturbed vegetation surrounding the spill site to document presence of non-resident plant species that may have migrated outside the previously revegetated areas. No non-resident species were identified at or in the immediate vicinity of the spill sites in 2021.

## Red Dog Tundra Working Group

The RDTWG was established in 2019 to invite agencies and communities to discuss best management practices related to coordinating cleanup activities and rehabilitating spill sites along the DMTS. Representatives from the following are involved in the RDTWG: NPS, U.S. Environmental Protection Agency (EPA), U.S. Army Corps of Engineers, Alaska Department of Natural Resources (DNR), U.S. Coast Guard, Northern Alaska Environmental Center, Kivalina City Council, Kivalina IRA, NANA, NANA/Lynden, and Alaska Industrial Development and Export Authority.

The RDTWG met in July 2021 at RDO; the meeting included a tour to visit several historical spill sites that were previously cleaned up according to DEC guidelines using excavators to remove concentrate from the tundra. RDTWG members present were Oral Kuki Hawley (Kivalina IRA), Theodore Booth (Kivalina IRA), Kimberley Maher (DEC-SPAR), Alyssa Millard (DNR), and Peter Neitlich (NPS), and Sue Bishop and Tim Cater (ABR Restoration Ecologists). Cleanup levels had been achieved at these sites, and instead the focus of the meeting was to design a rehabilitation study to learn more about tundra rehabilitation methods in the northwest Arctic.

The rehabilitation study was proposed to learn more about tundra rehabilitation following land disturbance in the northwest Arctic. The goal of the study is to identify the most effective and efficient rehabilitation techniques for concentrate spill sites along the DMTS road. With the exception of one diesel spill site, the sites selected for the rehabilitation study included locations where historical spills had occurred; note all spill were cleaned up using excavation and meet cleanup standards. The rehabilitation study was designed with input from all members of the RDTWG present at the July 2021 meeting. The study will examine revegetation success following treatments at three types of sites: 1) excavated and unvegetated spill sites with seed and fertilizer treatments; 2) minimally excavated spill sites with the surrounding tundra treated

with fertilizer; and 3) previously rehabilitated sites using either seed and fertilizer or tundra sod. The rehabilitation study will also include ongoing monitoring on historical spill sites that have been successfully reclaimed, as these sites may improve our understanding of the rehabilitation process over long periods.

#### Concentrate Spills in 2021

There were no concentrate spills along the DMTS in 2021.

## Worker Dust Protection Actions

The Worker Dust Protection Plan was prepared in response to RMP Objective #7: *Protect worker health*. To achieve this objective, the Worker Dust Protection Plan was developed with the goal: "To minimize worker exposure to fugitive dust, provide ongoing monitoring of exposure, and ensure a comprehensive communication system."

Safety is a core value for Teck, and the company is committed to providing leadership and resources for managing safety and health. Accordingly, the company has developed Environment, Health, Safety and Community Management Standards applicable to its operations worldwide. In addition, Teck has a comprehensive Occupational Safety and Health Program tailored specifically to RDO to protect worker health. The program complements the corporate standards and is designed to manage all aspects of workplace safety and health, including worker dust protection. The Worker Dust Protection Plan ties in closely with the existing health and safety programs at the mine that are overseen by the Red Dog medics.

The Blood Lead Biological Monitoring Program is for personnel who work in areas that meet or exceed Occupational Safety and Health Administration action levels for lead exposure. The frequency of monitoring for an individual worker is dependent on previous blood lead level history. The higher an individual's blood lead levels, the more frequent that worker will be monitored. The overall goal is to keep worker lead exposure as low as possible through engineering controls, administrative controls, and use of personal protective equipment and proper hygiene practices.

Worksite blood lead monitoring was conducted in 2021 by the Red Dog Human Resources Medical Department. Blood lead level testing is performed for all employees on a regular basis. The Red Dog blood lead standards are as follows:

- 0-20 mcg/dL: continue testing employee every 6 months
- 20.1–25 mcg/dL: test employee every 3 months and review protocol for use and cleaning of respirator
- 25.1–40 mcg/dL: continue testing employee every month and provide employee counseling by supervisors on the importance of proper respirator usage and the need for best practices regarding personal hygiene
- >40 mcg/dL: relocate employee from normal work area to an area with lower lead exposure until blood lead levels are 25 mcg/dL or less.

In 2021, all female employees had blood lead levels below 15 mcg/dL. In 2021, seven males had blood lead levels in the 20–24 mcg/dL range, and one male had blood lead levels in the 35–39 mcg/dL range. For these individual cases, the supervisor discussed elevated lead levels and reviewed work habits with the employee, including cleaning of filter mask, appropriate filter change protocols, and personal hygiene habits. No Teck Red Dog workers were removed from the job due to elevated blood lead levels.

## **Uncertainty Reduction Actions**

The Uncertainty Reduction Plan follows from RMP Objective #5: *Conduct research or studies to reduce uncertainties in the assessment of effects to humans and the environment*. To achieve this objective, the Uncertainty Reduction Plan was developed with the goal: "To identify and prioritize prospective research or studies to reduce uncertainties in the assessment of effects of fugitive dust to humans and the environment." In 2020, the white paper titled "*Effect of Time on Bioaccessibility of Aluminum and Barium in Arctic Soils*" was shared as part of the annual report. Comments were provided by DEC. The comments received are shared in Appendix A of this annual report, along with the responses. The review is greatly appreciated.

## **Monitoring Actions**

The Monitoring Plan (Exponent 2014) is intended to achieve the following RMP objectives:

- Objective 1: Continue reducing fugitive metals and dust emissions (This objective is indirectly addressed through monitoring, to verify effectiveness of operational dust control measures.).
- Objective 3: Verify continued safety of caribou, other representative subsistence foods, and water.
- Objective 4: Monitor conditions in various ecological environments and habitats and implement corrective measures when action levels are triggered.
- Objective 6: Improve collaboration and communication among all stakeholders to increase the level of awareness and understanding of fugitive dust issues.

To achieve these objectives, the Monitoring Plan was developed with the goal: "To monitor changes in dust emissions and deposition over time and space, using that information to: 1) assess the effectiveness of operational dust control actions, 2) evaluate the effects of the dust emissions on the environment and on human and ecological exposure, and 3) trigger additional actions where necessary."

Actions included in the Monitoring Plan were developed from priorities identified during development of the RMP, with input from local stakeholders, technical experts, and state and federal regulatory agencies. This section presents the results of the Monitoring Plan actions implemented during 2021. An overview of the components of the monitoring program along with the frequency of monitoring is shown in Figure 2. A map-based illustration of the risk management monitoring program components and monitoring stations and sites is shown in Figure 3.

## Monitoring Programs for DEC Oversight

The marine sediment and soil monitoring programs are ongoing and require DEC oversight, and the monitoring results are also used for trend analysis at RDO. Sediment monitoring is conducted every two years, and soil monitoring occurs once every three years. Both soil and sediment monitoring occurred in 2020, and those results were included in the 2020 RMP annual report. The next sediment monitoring event is scheduled for 2022 and the next soil monitoring even is scheduled for summer 2023.



#### **Operational Monitoring**

#### U.S. EPA Method 22—Visible Emissions Evaluation

Visible emissions evaluations (VEEs) were conducted as required for the Title V of the Clean Air Act operating permit at the mine. VEE monitoring occurs at multiple locations within the mine boundary and at the port. Along all unpaved roads, including the DMTS haul road, calcium chloride and/or water is used to control fugitive dust emissions when the road surfaces are not frozen or when the road surfaces do not exhibit visible surface moisture. To verify the effectiveness of these control measures, VEE observations are conducted daily when road surfaces are dry and not frozen. If dust is visibly present for more than 2 minutes on the unpaved road surfaces, additional calcium chloride or water is applied as soon as practicable and VEE monitoring is repeated to verify fugitive emissions are no longer present. All VEE readings that are required under the Title V permit are submitted twice a year to DEC within the Title V Facility Operating Report.

#### Partisol (Formerly TEOM) Source Monitoring

Historically at Red Dog Mine and Port, monitoring equipment known as tapered element oscillating microbalance (TEOM) instruments have been used to provide real-time total suspended particulate (TSP) information for mine operators at RDO. The instruments have been used since 2002 to monitor air quality at four locations near sources within the mine and port (Figures 3, 4, 5). However, the TEOM monitoring equipment was no longer supported by ThermoScientific, meaning parts and service for the instrumentation could no longer be procured. To provide for a smooth transition, beginning in 2019, Partisol (Model 2025i) instruments were placed in the same location as the TEOMs (Figures 3, 4, 5) to begin collecting data. In 2021, the TEOMs were retired and the Partisol (Model 2025i) instruments were used exclusively to obtain real-time source monitoring data.

Similar to the TEOMs, the Partisol instruments produce real-time measurements of dust in air and collect discrete samples, which are then analyzed to provide airborne metals concentrations. Measurements are reported as TSP, and zinc and lead concentrations are reported as TSP-Zn and TSP-Pb, respectively. Partisol instruments are operated continuously<sup>3</sup> to measure real-time TSP. Filters are used to collect TSP over 24-hour periods every third day at the mine and every sixth day at the port, and are then analyzed for TSP-Zn and TSP-Pb. The Mine Partisol samplers are located downwind of the pit and crusher at the Personnel Accommodations Complex (PAC), and at the main tailings dam (T-Dam) downwind of the tailings beach, mill, and other facilities (Figure 4). The Port Partisol samplers are located downwind of the CSBs and in the lagoon area downwind of the concentrate conveyor (Figure 5).

Statistical Trend Analysis for Partisol Data. Statistical testing methods were used to evaluate whether the Partisol (spliced together with prior TEOM) data sets exhibit statistically significant temporal trends in metals concentrations. The Seasonal Mann-Kendall trend test is a nonparametric method to investigate temporal trends in time series containing substantial seasonal variability. For this analysis, the real-time data were summarized on a monthly basis. Seasonal trend tests were conducted using monthly means and monthly upper limit 95th percentile concentrations to evaluate both average conditions and measures of the upper limits over the past four years (beginning of 2018 to end of 2021). Results of the statistical trend tests for real-time data (lead and zinc concentrations) in four locations (Mine PAC, Mine T-Dam, Port CSB, and Port Lagoon) are summarized in Table 1.

The calculated monthly averages for TSP-Pb and TSP-Zn concentrations are shown on Figures 6 and 7, respectively, for all four mine and port Partisol/TEOM locations. The concentrations of lead and zinc at the mine area are typically higher than those at the port area (Figures 6 and 7, respectively). At the mine and port, lead and zinc concentrations were typically lowest in summer months (the months with higher humidity and more road watering for dust control), and highest in winter months (the coldest, driest, and lowest humidity months, when road watering is not possible because of freezing conditions).

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<sup>&</sup>lt;sup>3</sup> Occasional system upsets do occur as a result of weather or equipment failure. Partisol readings are monitored frequently so that system upsets are noted and corrected as soon as possible. Missing or unusable data are noted in the raw data files and are not used in statistical trend evaluations.

- Mine PAC Results. The Mine PAC results show significant increasing trends in mean and upper limit 95<sup>th</sup> percentile concentrations for lead and zinc (Figures 6 and 7, Table 1).
- Mine T-Dam Results. There was a significant decreasing trend in both mean and upper limit 95<sup>th</sup> percentile lead and zinc concentrations over the past four years at the Mine T-Dam (Figures 6 and 7, Table 1).
- **Port CSB Results.** There was a significant decreasing trend in both mean and upper limit 95<sup>th</sup> percentile lead concentrations at the Port CSB over the past four years (Figure 6, Table 1). In contrast, there was a significant increasing trend in both mean and upper limit 95<sup>th</sup> percentile zinc concentrations over the past four years (Figure 7, Table 1).
- **Port Lagoon Results.** The Port Lagoon Partisol and TEOM results show significant increasing trends in mean and upper limits 95<sup>th</sup> percentile concentrations for lead and zinc over the past four years (Figures 6 and 7, Table 1).

**Real Time Alarm System Monitoring.** Real-time SHARP (synchronized hybrid ambient realtime particulate) monitor (ThermoScientific 5030i SHARP) data are used to monitor for high dust events so that mine activities can be modified (where possible) to reduce dust levels. When air quality measurements exceed a warning level or an alarm level, the alarm status is displayed on the Red Dog weather intranet web page to notify personnel within the Mine Operations and Environmental departments to take corrective action. Examples of these corrective actions include applying water on the roads or stockpiles, or shutting down loading operations during windy conditions.

#### Road Surface Monitoring

Loose fine materials that may be subject to airborne transport are sampled from the road surface at eight locations every two months. From the mine site to the port, the eight-road surface monitoring station locations are:

- Mine CSB (near exit from truck loading portion of CSB)
- The Y (near the back dam, between the CSB and the Airport)

- Airport
- MS-13 (former material site where road crosses the mine boundary)
- MS-9 (material site between the mine and CAKR)
- R-Boundary (northern boundary of CAKR)
- MS-2 (material site just inside the northern boundary of the port)
- Port CSB Track (road near exit from truck unloading building at the port CSBs).

Samples are analyzed onsite using a portable XRF analyzer to determine lead, zinc, and cadmium concentrations within road surface materials. The "Mine CSB" and "The Y" stations (inside the operational mine boundary) generally exhibit higher metals concentrations and are managed so as to reduce tracking of metals toward the port with a traffic plan and truck wash that is operational during warmer months.

During most recent four-year period (2018–2021), statistical analyses indicate lead, zinc, and cadmium concentrations in road fugitive dust samples from the mine, port, and road have either significantly decreased or show no significant trend (Figures 8, 9, and 10; Table 2). When each location is examined separately, the same was true, no significant increases, except directly outside the Mine CSB, where zinc (but not lead or cadmium) concentrations had significantly increased over the past four years (Table 2).

If measured road surface concentrations at stations outside the mine boundary exceed Arctic Zone Industrial Cleanup Levels for lead, zinc, or cadmium (800, 41,100, and 110 mg/kg respectively<sup>4</sup>) for more than two consecutive sampling periods, that road section is to be remediated and resurfaced as described in the Remediation Plan (Exponent 2011). No additional road remediation was required during 2021 because results for stations outside the mine and port boundaries did not exceed the Arctic Zone Industrial Cleanup Levels.

<sup>&</sup>lt;sup>4</sup> Cleanup levels according to 18 AAC 75.341, as revised in 2008 (available on the internet at <u>https://dec.alaska.gov/spar/csp/docs/75mas\_art3.pdf</u>). Note that the cadmium and zinc cleanup level would be lower, at 79 and 30,400 mg/kg, if the zone were considered to be the "Under 40-inch Zone" by DEC, which is a function of the definitions at 18 AAC 75.990.

#### **Dustfall Jar Monitoring**

Dustfall jars are passive continuous collectors used for measuring dust deposition; samples are collected every two months at all locations. Approximately 86 dustfall stations are located around the mine, port, and DMTS road (Figure 3), as follows:

- At the mine, approximately 34 jars are placed in locations around the facilities.
- Along the DMTS road, 12 dustfall jars are located at three stations, each with four dustfall jars, two on either side of the road. The DMTS road stations are collocated with road surface sampling stations near the port boundary, the CAKR northern boundary, and midway between CAKR and the mine. The dustfall jars are located approximately 100 m from the shoulder of the DMTS, with 100 m between them, oriented parallel to the road.
- At the port, 38 jars are placed roughly in a rectangular grid throughout the area.
- An additional two jars are considered reference stations, one upwind of the road near Evaingiknuk Creek, and another near the Wulik River, to the north of the mine operation.

<u>Statistical Trend Analysis for Dustfall Jar Data.</u> Temporal trends in deposition rates or metals concentrations in dustfall jar data were evaluated using seasonal trend tests conducted with bimonthly mean and 95th percentiles (method same as discussed above in TEOM section).

- Lead. No statistically significant trends were identified in lead deposition rates during the most recent four-year monitoring period at the mine, along the DMTS road, or port (Table 3). Also, no statistically significant trends in lead concentrations were detected along the DMTS road or at the port. However, lead concentrations at the mine dustfall stations did significantly increase over the past four years. Time series plots of lead concentrations and dustfall deposition rates are presented in Figures 11 and 12, respectively.
- **Zinc.** No statistically significant trends were identified in zinc deposition rates during the most recent four-year monitoring period at the mine, along the DMTS road, or port (Table 3). At the mine and port, significant increasing trends in zinc concentrations were

detected (Table 3). No significant trends in zinc concentrations were detected along the DMTS road.

• **Total Solids.** No statistically significant trends were identified in total solids deposition rates during the most recent four-year monitoring period at the mine, along the DMTS road, or port (Table 3). For total solids, the deposition rates have been stable with no statistically significant trends identified at any location over the most recent four-year period (Table 3). Time series plots of total solids dustfall rates are presented in Figure 15.

#### Caribou Tissue Monitoring

Red Dog Mine is located within the normal annual range of the Western Arctic Herd of caribou. Surveys of caribou tissue metals concentrations have been conducted periodically since 1984 by the Alaska Department of Fish and Game and these data provide baseline information against which more current studies may be compared. Previous caribou tissue monitoring events near Red Dog were completed in 2002 and 2009. The next caribou tissue monitoring (meat, kidney, liver) event is currently scheduled for 2025; therefore, there are no results to present for 2021.

#### Summary of Monitoring Results

Dust monitoring data from the Partisol and TEOM air samplers, road surface samples, and the dustfall jars were statistically evaluated to assess the current trends over the most recent fouryear period. A summary of statistical trend analysis results for Partisol and TEOM, road surface, and dustfall jar monitoring programs is presented in Table 4. Table 4 provides an at-a-glance overview of results of dust monitoring programs.

Road dust samples collected at locations at the mine, road, and port have shown either significant decreases or no statistically significant change over the past four-year period for lead, zinc, and cadmium (2018–2021). Dustfall jars along the road and also show no significant changes over the past four-year period for lead and zinc concentrations and in rates of dustfall deposition. The Partisol data collected near the mine tailings dam show significant decreases in lead and zinc concentrations over the past four-year period, but in contrast, the mine Partisol

data shows significant increases for lead and zinc concentrations. Similarly, dustfall jars at the mine indicated significant increases in lead and zinc over the past four years. A summary of all results is shown in Table 4.

The goal of the Red Dog Operations Fugitive Dust Risk Management Program [Minimize risk to human health and the environment surrounding the DMTS and outside the Red Dog Mine boundary over the life of the mine] is to ensure that dust levels remain low using the risk management actions discussed in this 2021 report. These monitoring programs provide Red Dog with data at a high frequency (data collection ranging from every other day to once every two months) and within areas located right near the operations. The intent is to use this information to inform Red Dog where the most attention will be needed and to avoid increases beyond the Red Dog Mine and Port ambient air/solid waste permit boundaries. The 2021 results indicate some increases noted over the last four-year period, indicating that additional attention to potential dust controls will need to be considered in 2022.



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## Appendix A

## Figures

|   | Fugitive Dust<br>Risk Management Plan |                                 |                                  |                    |                               |                                |  |  |
|---|---------------------------------------|---------------------------------|----------------------------------|--------------------|-------------------------------|--------------------------------|--|--|
| RISK MANAGEMENT<br>OBJECTIVES   | Communication<br>Plan                 | Dust Emission<br>Reduction Plan | Remediation/<br>Reclamation Plan | Monitoring<br>Plan | Uncertainty<br>Reduction Plan | Worker Dust<br>Protection Plan |  |  |
| 1. Continue reducing fugitive metals emissions and dust emissions   |                                       |                                 |                                  |                    |                               |                                |  |  |
| <ol> <li>Conduct remediation or reclamation in selected areas</li> </ol>  |                                       |                                 |                                  |                    |                               |                                |  |  |
| <b>3.</b> Verify continued safety of caribou, other representative subsistence foods, and water   |                                       |                                 |                                  |                    |                               |                                |  |  |
| 4. Monitor conditions in various<br>ecological environments and<br>habitats, and implement<br>corrective measures when action<br>levels are triggered |                                       |                                 |                                  |                    |                               |                                |  |  |
| <ol> <li>Conduct research or studies to<br/>reduce uncertainties in the<br/>assessment of effects to humans<br/>and the environment</li> </ol>        |                                       |                                 |                                  |                    | -                             |                                |  |  |
| 6. Improve communication and collaboration among all stakeholders   |                                       |                                 |                                  |                    |                               |                                |  |  |
| 7. Protect worker health  |                                       |                                 |                                  |                    |                               |                                |  |  |
|   | Implementation p                      | lan directly addresses of       | ojective                         |                    |                               |                                |  |  |

Implementation plan indirectly addresses objective

Figure 1. Risk management objectives and associated implementation plans



Figure 2. Monitoring timeline with program frequencies


Figure 3. Overview of risk management monitoring programs

# October 2019



Figure 4. Mine Partisol/TEOM locations



Figure 5. Port Partisol/TEOM locations



Figure 6. TEOM Lead Concentration plots (all years)



Figure 7. TEOM Zinc Concentration plots (all years)





Figure 9. Road Surface Lead Concentrations (all years)



Figure 10. Road Surface Zinc Concentrations (all years)



Figure 11. Dustfall Jars Lead Conc. plots (all years)



Figure 12. Dustfall Jars Lead Rate plots (all years)



Figure 13. Dustfall Jars Zinc Conc. plots (all years)



Figure 14. Dustfall Jars Zinc Rate plots (all years)



Figure 15. Dustfall Jars Solids Rate plots (all years)

# Tables

Table 1. Partisol and TEOM concentration statistical trend analysis (Seasonal Mann-Kendall Trend Test)

|                   | Concentration (µg/m <sup>3</sup> ) |          |                                 |  |  |  |
|-------------------|------------------------------------|----------|---------------------------------|--|--|--|
| LEAD              | tau statistic                      | p value  | significant trend? <sup>a</sup> |  |  |  |
| Mine PAC          | -0.298                             | 0.000425 | Yes; Increasing                 |  |  |  |
| Mine T-Dam        | -0.366                             | 2.35E-05 | Yes; Decreasing                 |  |  |  |
| Port CSB          | -0.361                             | 2.69E-05 | Yes; Decreasing                 |  |  |  |
| Port Lagoon       | -0.319                             | 0.000282 | Yes; Increasing                 |  |  |  |
| Port CSB & Lagoon | -0.36                              | 2.06E-05 | Yes; Decreasing                 |  |  |  |

For 1/2018 - 12/2021; Mean Concentration

| 7100              | Concentration (µg/m <sup>3</sup> ) |          |                                 |  |  |  |
|-------------------|------------------------------------|----------|---------------------------------|--|--|--|
| ZINC              | tau statistic                      | p value  | significant trend? <sup>a</sup> |  |  |  |
| Mine PAC          | -0.28                              | 0.000953 | Yes; Increasing                 |  |  |  |
| Mine T-Dam        | -0.366                             | 2.35E-05 | Yes; Decreasing                 |  |  |  |
| Port CSB          | -0.331                             | 0.000116 | Yes; Increasing                 |  |  |  |
| Port Lagoon       | -0.257                             | 0.00349  | Yes; Increasing                 |  |  |  |
| Port CSB & Lagoon | -0.317                             | 0.000181 | Yes; Increasing                 |  |  |  |

<sup>a</sup> Significant at p<0.05/2 (i.e., p<0.025 with Bonferroni adjustment because multiple [2] related hypotheses are tested).

## For 1/2018 - 12/2021; Top 95% Concentration

|                   | Concentration (µg/m <sup>3</sup> ) |          |                                 |  |  |  |
|-------------------|------------------------------------|----------|---------------------------------|--|--|--|
| LLAD              | tau statistic                      | p value  | significant trend? <sup>a</sup> |  |  |  |
| Mine PAC          | -0.292                             | 0.000559 | Yes; Increasing                 |  |  |  |
| Mine T-Dam        | -0.373                             | 1.67E-05 | Yes; Decreasing                 |  |  |  |
| Port CSB          | -0.364                             | 0.000023 | Yes; Decreasing                 |  |  |  |
| Port Lagoon       | -0.303                             | 0.00059  | Yes; Increasing                 |  |  |  |
| Port CSB & Lagoon | -0.366                             | 1.48E-05 | Yes; Decreasing                 |  |  |  |

| 71NC              | Concentration (µg/m <sup>3</sup> ) |          |                                 |  |  |
|-------------------|------------------------------------|----------|---------------------------------|--|--|
| ZINC              | tau statistic                      | p value  | significant trend? <sup>a</sup> |  |  |
| Mine PAC          | -0.255                             | 0.00261  | Yes; Increasing                 |  |  |
| Mine T-Dam        | -0.373                             | 1.67E-05 | Yes; Decreasing                 |  |  |
| Port CSB          | -0.305                             | 0.000381 | Yes; Increasing                 |  |  |
| Port Lagoon       | -0.264                             | 0.0027   | Yes; Increasing                 |  |  |
| Port CSB & Lagoon | -0.304                             | 0.000321 | Yes; Increasing                 |  |  |

<sup>a</sup> Significant at p<0.05/2 (i.e., p<0.025 with Bonferroni adjustment because multiple [2] related hypotheses are tested).

Table 2. Road surface concentration statistical trend analysis (Seasonal Mann-Kendall Trend Test)

|                       |             | Concer        | ntration (pp | m)                              |
|-----------------------|-------------|---------------|--------------|---------------------------------|
| LEAD                  |             | tau statistic | p value      | significant trend? <sup>a</sup> |
| Mine <sup>b</sup>     |             | -0.323        | 0.0128       | Yes; Decreasing                 |
| Port                  |             | -0.214        | 0.0874       | No                              |
| Road                  |             | -0.333        | 0.0069       | Yes; Decreasing                 |
| Mine CSB (Mine)       |             | 0.102         | 0.408        | No                              |
| The Y (Mine)          | 2018 - 2021 | -0.333        | 0.00789      | Yes; Decreasing                 |
| Airport (Mine)        |             | -0.26         | 0.045        | No                              |
| MS-13 (Mine/Road)     |             | -0.277        | 0.0275       | No                              |
| MS-9 (Road)           |             | -0.238        | 0.0537       | No                              |
| R-Boundary (Road)     |             | -0.374        | 0.00243      | Yes; Decreasing                 |
| MS-2 (Port)           |             | -0.343        | 0.00624      | Yes; Decreasing                 |
| Port CSB Track (Port) |             | -0.163        | 0.194        | No                              |

For 1/2018 - 12/2021; Mean Concentration:

| ZINC                  |             | Concer        | ntration (ppi | m)                              |
|-----------------------|-------------|---------------|---------------|---------------------------------|
| ZINC                  |             | tau statistic | p value       | significant trend? <sup>a</sup> |
| Mine <sup>b</sup>     |             | 0.228         | 0.0781        | No                              |
| Port                  |             | -0.171        | 0.171         | No                              |
| Road                  | 2018 - 2021 | -0.361        | 0.00348       | Yes; Decreasing                 |
| Mine CSB (Mine)       |             | 0.714         | 7.09E-09      | Yes; Increasing                 |
| The Y (Mine)          |             | -0.319        | 0.011         | Yes; Decreasing                 |
| Airport (Mine)        |             | -0.26         | 0.045         | No                              |
| MS-13 (Mine/Road)     |             | -0.262        | 0.0365        | No                              |
| MS-9 (Road)           |             | -0.265        | 0.0315        | No                              |
| R-Boundary (Road)     |             | -0.374        | 0.00243       | Yes; Decreasing                 |
| MS-2 (Port)           |             | -0.343        | 0.00624       | Yes; Decreasing                 |
| Port CSB Track (Port) |             | -0.0922       | 0.462         | No                              |

|                       |             | Concer        | ntration (pp | m)                              |
|-----------------------|-------------|---------------|--------------|---------------------------------|
| CADIVITOIN            |             | tau statistic | p value      | significant trend? <sup>a</sup> |
| Mine <sup>b</sup>     |             | -0.0133       | 0.931        | No                              |
| Port                  |             | -0.0824       | 0.578        | No                              |
| Road                  |             | -0.156        | 0.283        | No                              |
| Mine CSB (Mine)       |             | -0.156        | 0.283        | No                              |
| The Y (Mine)          | 2018 - 2021 | -0.106        | 0.474        | No                              |
| Airport (Mine)        |             | -0.0133       | 0.931        | No                              |
| MS-13 (Mine/Road)     |             | -0.106        | 0.474        | No                              |
| MS-9 (Road)           |             | -0.111        | 0.443        | No                              |
| R-Boundary (Road)     |             | -0.156        | 0.283        | No                              |
| MS-2 (Port)           |             | -0.0353       | 0.812        | No                              |
| Port CSB Track (Port) |             | -0.106        | 0.474        | No                              |

<sup>a</sup>Significant at p<0.05/3 (i.e., p<0.017 with Bonferroni adjustment because multiple [3] related hypotheses are tested)

<sup>b</sup>MS-13 included in Mine

Table 3. Dustfall rate and concentrations statistical trend analysis (Seasonal Mann-Kendall Trend Test)

|           | Dustfall Desposition Rate (mg/m <sup>2</sup> /day) |         |                                 | Concentration (mg/kg-total solid) |           |                                 |
|-----------|--|---------|---------------------------------|-----------------------------------|-----------|---------------------------------|
| LLAD      | tau statistic                                      | p value | significant trend? <sup>a</sup> | tau statistic                     | p value   | significant trend? <sup>a</sup> |
| Mine      | -0.0676  | 0.467   | No                              | 0.417                             | 0.0000072 | Yes; Increasing                 |
| DMTS Road | -0.0396  | 0.67    | No                              | 0.0303                            | 0.744     | No                              |
| Port      | 0.0207   | 0.828   | No                              | 0.155                             | 0.103     | No                              |
| Reference | 0.0526   | 0.589   | No                              | 0.28                              | 0.00406   | Yes; Increasing                 |

For 1/2018 - 12/2021; Mean Deposition Rate and Concentration:

| 71010     | Dustfall Desposition Rate (mg/m <sup>2</sup> /day) |         |                                 | Concentration (mg/kg-total solid) |          |                                 |
|-----------|--|---------|---------------------------------|-----------------------------------|----------|---------------------------------|
| ZINC      | tau statistic                                      | p value | significant trend? <sup>a</sup> | tau statistic                     | p value  | significant trend? <sup>a</sup> |
| Mine      | -0.0338  | 0.718   | No                              | 0.812                             | 0        | Yes; Increasing                 |
| DMTS Road | -0.035   | 0.707   | No                              | 0.021                             | 0.821    | No                              |
| Port      | 0.0259   | 0.786   | No                              | 0.285                             | 0.00282  | Yes; Increasing                 |
| Reference | 0.0416   | 0.67    | No                              | 0.324                             | 0.000873 | Yes; Increasing                 |

|              | Dustfall Desposition Rate (mg/m <sup>2</sup> /day) |         |                                 |  |  |
|--------------|--|---------|---------------------------------|--|--|
| TOTAL SOLIDS | tau statistic                                      | p value | significant trend? <sup>a</sup> |  |  |
| Mine         | -0.0396  | 0.67    | No                              |  |  |
| DMTS Road    | -0.0443  | 0.634   | No                              |  |  |
| Port         | 0.0104   | 0.914   | No                              |  |  |
| Reference    | 0.0305   | 0.754   | No                              |  |  |

<sup>a</sup> Significant at p<0.05/3 (i.e., p<0.017 with Bonferroni adjustment because multiple [3] related hypotheses are tested).

#### For 1/2018 - 12/2021; Top 95% Deposition Rate and Concentration:

|           | Dustfall Deposition Rate (mg/m <sup>2</sup> /day) |         |                                 | Concentration (mg/kg-total solid) |            |                                 |
|-----------|---|---------|---------------------------------|-----------------------------------|------------|---------------------------------|
| LEAD      | tau statistic                                     | p value | significant trend? <sup>a</sup> | tau statistic                     | p value    | significant trend? <sup>a</sup> |
| Mine      | -0.0676   | 0.467   | No                              | 0.622                             | 2.17E-11   | Yes; Increasing                 |
| DMTS Road | -0.049  | 0.599   | No                              | 0.166                             | 0.0751     | No                              |
| Port      | -0.00518  | 0.957   | No                              | 0.394                             | 0.0000368  | Yes; Increasing                 |
| Reference | 0.0582  | 0.55    | No                              | 0.438                             | 0.00000691 | Yes; Increasing                 |

| ZINC      | Dustfall Deposition Rate (mg/m <sup>2</sup> /day) |         |                                 | Concentration (mg/kg-total solid) |           |                                 |
|-----------|---|---------|---------------------------------|-----------------------------------|-----------|---------------------------------|
| LINC      | tau statistic                                     | p value | significant trend? <sup>a</sup> | tau statistic                     | p value   | significant trend? <sup>a</sup> |
| Mine      | -0.0338   | 0.718   | No                              | 0.831                             | 0         | Yes; Increasing                 |
| DMTS Road | -0.0396   | 0.67    | No                              | 0.138                             | 0.139     | No                              |
| Port      | 0.0311  | 0.745   | No                              | 0.611                             | 1.48E-10  | Yes; Increasing                 |
| Reference | 0.036   | 0.712   | No                              | 0.38                              | 0.0000972 | Yes; Increasing                 |

|              | Dustfall Deposition Rate (mg/m <sup>2</sup> /day) |         |                                 |  |  |
|--------------|---|---------|---------------------------------|--|--|
| TOTAL SOLIDS | tau statistic                                     | p value | significant trend? <sup>a</sup> |  |  |
| Mine         | -0.0676   | 0.467   | No                              |  |  |
| DMTS Road    | -0.0396   | 0.67    | No                              |  |  |
| Port         | -0.00518  | 0.957   | No                              |  |  |
| Reference    | 0.0305  | 0.754   | No                              |  |  |

<sup>a</sup> Significant at p<0.05/3 (i.e., p<0.017 with Bonferroni adjustment because multiple [3] related hypotheses are tested).

Table 4. Summary of dust monitoring trends

|   |   |  |  |   |                        | For mo     | ost recent 4              | years (201                           | .8-2021)   |                        |              |                               |                                   |                           |            |
|---|---|--|--|---|------------------------|------------|---------------------------|--------------------------------------|--|------------------------|--------------|-------------------------------|-----------------------------------|---------------------------|------------|
|   | Road Sur                                      | rface (Conce                                   | ntration)                                      | Paritsol & TEOM   |                        |            | (Air Concentration)       |                                      | Dustfall Jars (Concentration and Deposition Rate)        |                        |              |                               |                                   |                           |            |
| Location<br>and<br>Measure  |   | Mean   |  | Location and<br>Measure                                   | M                      | ean        | 95 <sup>th</sup> Pe       | rcentile                             | Location and<br>Measure                                  |                        | Mean         |                               | 95                                | ith Percent               | ile        |
| wiedsure  | Pb  | Zn   | Cd   |   | Pb                     | Zn         | Pb                        | Zn                                   |  | Pb                     | Zn           | Solids                        | Pb                                | Zn                        | Solids     |
| Mine <sup>b</sup><br>(Conc.)  | И   |  | _  | Mine PAC<br>(Conc.)<br>Mine TDam                          | 7                      | Ζ Σ        | Z N                       | <b>N</b>                             | Mine (Conc.)   |                        | ~            | a                             | <b>N</b>                          | <b>N</b>                  | а          |
|   |   |  |  | (Conc.)   |                        |            |                           |                                      |  |                        |              |                               |                                   |                           |            |
| Road  | N   | N  |  |   |                        |            |                           |                                      | Road (Conc.)   | _                      |              | а                             |                                   |                           | а          |
| (conc.)   |   |  |  |   |                        |            |                           |                                      | Road<br>(Rate)   |                        |              |                               | _                                 | _                         | _          |
| Port<br>(Conc.)   | —   |  |  | Port CSB<br>(Conc.)                                       | И                      | 7          | И                         | 7                                    | Port (Conc.)   |                        | 7            | а                             | 7                                 | 7                         | а          |
|   |   |  |  | Port Lagoon<br>(Conc.)                                    | 7                      | 7          | 7                         | 7                                    | Port (Rate)  | _                      | —            | —                             |                                   | —                         |            |
|   |   |  |  | Port CSB &<br>Lagoon                                      | И                      | 7          | И                         | 7                                    |  |                        |              |                               |                                   |                           |            |
|   |   |  |  |   |                        |            |                           |                                      | Reference<br>(Conc.)<br>Reference<br>(Rate)              | ⊼                      | ⊼            | a                             | <b>7</b>                          | <b>N</b>                  | а          |
| <sup>a</sup> Concentrati  | on is not eva                                 | luated for so                                  | ids, because                                   | total solids is the                                       | e entire               |            | 1. Results a              | e presente                           | d for statistical tes                                    | ting using d           | ata from the | past four year                | rs.                               |                           |            |
| sample mass<br><sup>b</sup> MS-13 inclue<br><sup>c</sup> No Cadmiun | ded in Mine<br>n data since :                 | 2018 so trenc                                  | ls have not c                                  | hanged  |                        |            |                           |                                      |  |                        |              |                               |                                   |                           |            |
| Notes:  | Indicates no<br>tested (tren                  | statistically s<br>d is FLAT).                 | ignificant ch                                  | ange over time p  | eriod                  | 7          | Indicates a speriod teste | statistically<br>ed (trend is l      | significant increas<br>JP).                              | e over time            | И            | Indicates a st<br>time period | tatistically sig<br>tested (trend | nificant dec<br>is DOWN). | rease over |
| TEOM = tape<br>Conc = air co<br>Rate = dustfa                       | red element<br>ncentration (<br>Il deposition | oscillating m<br>(TEOM air sar<br>rate based o | icrobalance<br>npling) or cc<br>n dustfall jai | (air sampling dev<br>oncentration in du<br>r measurements | ice)<br>ıstfall (dustf | fall jars) |                           | TDam = m<br>PAC = pers<br>CSB = conc | ine tailings dam<br>onnel accommod<br>centrate storage b | ations comp<br>uilding | blex         |                               |                                   |                           |            |

# Appendix A

IVBA Results, Response to Comments, and Raw Data Tables

#### 1 RESEARCH NOTE

# Effect of time and soil particle size on the in vitro bioaccessibility of aluminum and barium in Arctic tundra soils

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#### 7 Keywords

8 Bioaccessibility, bioavailability, metals, Arctic soils, tundra, aluminum, barium

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#### 11 Abbreviations

Al: aluminum, As: arsenic, Ba: barium, DMTS: DeLong Mountain Regional Transportation System, DTSC: Department of Toxic
 Substances Control, EPA: U.S. Environmental Protection Agency, RDO: Red Dog Operations, ICP-MS: inductively coupled plasma
 mass spectrometry, ICP-OES: inductively coupled plasma-optical emission spectroscopy, IVBA: in vitro bioaccessibility, ISO:
 International Organization for Standardization, ITRC: Interstate Technology Regulatory Council, QA: quality assurance, Pb: lead, Zn:
 zinc

17

# 18 Abstract

19 Red Dog Operations (RDO), located in the western Brooks Mountain Range in Northwest Alaska, is 20 one of the largest producing zinc mines in the world. The surrounding area is characterized by Arctic 21 tundra ecosystem, dominated by mixed shrub-sedge tussock tundra interspersed with low-lying, well-22 drained knolls that support a variety of lichen, forbs, and shrubs. Arctic tundra soils were sampled near 23 RDO to measure the concentrations and in vitro bioaccessibility (IVBA) of aluminum (Al) and barium 24 (Ba), which were indicated as potential contributors toward ecological risk in the RDO fugitive dust risk 25 assessment that was completed in 2007. Al and Ba concentrations and IVBA were measured for two soil 26 particle size fractions. The objectives were to evaluate changes in metals concentrations and 27 bioaccessibility between 2005 and 2018, and to assess whether the measured bioaccessibility in these 28 soils is affected by soil particle size. Results of this study showed little change in Ba soil concentrations 29 and decreases in Al concentrations over time. The IVBA of these metals was shown to be low and did not 30 substantially change between the two time periods. Soil particle size did not predictably affect either the

31 concentrations or the IVBA of these metals in tundra soils. The results of this study provide information 32 to fill potentially important data gaps in the literature related to the environmental fate and stability of 33 metals in soils in general, and from Arctic regions specifically.

# 34 Introduction

35 A large body of research exists regarding the environmental chemistry of metals in soils and their 36 bioavailability, and how such data can be used to inform human and ecological risk assessment. Such 37 research indicates that metals in soil can be present in forms that are not fully bioavailable, and in 38 particular, not as bioavailable as the forms of metals used in laboratory toxicity testing. This is generally 39 well established for metals and metalloids in mine waste where bioavailability is affected by the mineral 40 form of the metal, soil particle size, and by soil characteristics that can encapsulate or otherwise affect the 41 solubility of metals under physiological conditions (Ruby et al. 1999, Shock et al. 2007, U.S. EPA 2007a, 42 b; Menzie et al. 2008, 2009; ITRC 2017). This study presents the results of an investigation of the IVBA 43 of Al and Ba from soils collected from tundra locations near Red Dog Mine, which is located in a remote 44 tundra environment in northwest Alaska above the Arctic Circle. Al and Ba were studied because 45 previous assessments that assumed 100% bioavailability for these metals indicated potentially increased 46 risk from Al and Ba exposure for animals in the vicinity of the mine and associated transportation 47 corridor (Shock et al., 2007; Exponent, 2007).

Bioavailability studies are typically conducted in animals. However, animal ("in vivo") studies are expensive, time consuming, and may raise ethical concerns. Therefore, laboratory methods have been developed to provide more efficient and inexpensive mechanisms to estimate the oral bioavailability of metals in soils relative to soluble forms. In vitro approaches include many different methods, generally intended to mimic conditions of the gastrointestinal tract (i.e., physiologically based). These methods rely on laboratory extraction testing of soil to assess the fraction of total chemical released, and the results are referred to in the published literature as bioelution, bioaccessibility, and IVBA.

55 IVBA results are used to estimate the relative bioavailability of chemicals from soil. Relative 56 bioavailability is the ratio of the fraction of metal absorbed from soil compared to the fraction absorbed 57 from the exposure medium used in the toxicity studies that form the basis for the default reference dose 58 for estimating human toxicity (Schoof 2003) and the toxicity reference values (TRVs) that are used to 59 evaluate toxicity to ecological receptors (Shock et al. 2007).

Regulatory agencies recognize that soil-chemical interactions may result in the reduced
bioavailability of certain chemicals from soil and therefore adjustments are accepted to account for this in
the risk assessment process for both human and ecological receptors (U.S. EPA 1998, 2007a, 2007b,
2007c, 2017; ITRC 2017, ISO 2018, California DTSC 2020, Whitacre et al. 2017; Hawaii Department of
Health 2020; Health Canada 2017). Adjustments based on site-specific bioavailability are used to refine
the results of a risk assessment by reducing uncertainties in the exposure assessment and associated risk
characterization.

#### 67 Study Background

68 Red Dog Mine is an operation using conventional drill and blast mining methods, and includes a mill 69 where concentrates are produced through crushing and grinding. The mine is located on land owned by 70 NANA, a for-profit Alaska Native Corporation, and was developed under an innovative operating 71 agreement between NANA and the mine operator, Teck Alaska. The concentrates are trucked along a 52-72 mile gravel haul road from the mine site to the coast, and delivered to the Chukchi Sea port facility for 73 shipment to markets around the world. The haul road and the port collectively make up the DeLong 74 Mountain Regional Transportation System (DMTS), built by the Alaska Industrial Development and 75 Export Authority (AIDEA) and completed in 1989.

During the early 2000s, a comprehensive human health and ecological risk assessment was conducted for tundra areas surrounding the DMTS and RDO. The bioavailability of all metals except lead (Pb) was assumed to be 100%; for Pb, adjustments were made in the human health risk assessment to account for its site-specific bioavailability (Exponent 2007, Garry et al. 2020). The ecological risk assessment, which assumed metals were 100% bioavailable from soil, showed a potential for increased risk to small

mammals from exposure to Al and Ba, with incidental ingestion of soil being the primary exposure
pathway (Exponent 2007, Shock et al. 2007). To further refine the results and reduce uncertainties
associated with the risk assessment, the site-specific Al and Ba bioaccessibility was evaluated in 2005
using in vitro methods as described in Shock et al. (2007). The Shock et al. (2007) study showed the
bioavailability (as estimated by IVBA) of Al and Ba from soils was substantially less than the default
assumption of 100%. Therefore, actual potential for adverse effects from these metals was concluded to
be small given the highly conservative nature of the risk assessment (Exponent 2007).

88

The information in this brief communication is specifically focused on summarizing the results of follow up IVBA testing for a small set of Arctic tundra soils affected by fugitive dust from RDO. The objectives were to determine whether the bioaccessibility of Al and Ba in these samples differs as a function of soil particle size, and to determine if there have been changes in the concentrations and bioaccessibility of these metals over time. Soil sampling was conducted in 2018, with sampling locations selected to replicate the locations where soils were sampled previously in 2005. The IVBA of Ba and Al were measured for different soil particle size fractions.

96 To provide information on the bioavailability of Al and Ba for these Arctic tundra soil samples, IVBA 97 extractions of samples were conducted under the conditions specified by U.S. Environmental Protection 98 Agency (EPA) Method 1340. This method has been validated for quantitative use in predicting relative 99 oral bioavailability of Pb and arsenic (As) for human health risk assessment (U.S. EPA 2017). Other 100 investigations have also established that the acidic extraction conditions used in EPA Method 1340 also 101 generate data that are generally predictive of the bioavailability of cadmium (Cd), for which an 102 International Organization for Standardization (ISO) Standard has been developed (ISO 2018; Schroder et 103 al. 2003; Boros et al. 2017). Similarly, investigations by Health Canada indicate that a low pH extraction 104 method, such as EPA Method 1340, results in the most conservative estimate of IVBA for zinc (Zn) 105 (Koch et al. 2013). Therefore, while validated by U.S. EPA (2018) for Pb and As only, EPA Method 1340 106 can also provide screening information for the bioavailability of other metals (Menzie et al. 2008).

107 Although limited in scope to only a few available samples, this study provides information to fill data 108 gaps in the literature that have been identified by several researchers, including gaps in information on 109 metals in Arctic environments and the stability of soil-chemical interactions over time (Rudnika-Kepa and 110 Zaborska 2021, Fernandez-Llamazares 2019, Camenzuli et al. 2014, Darrouzet-Nardi 2018, Sloan et al. 111 1997, Violante et al. 2010, Rieuwerts et al. 1998, Liang et al. 2016, Bradham et al. 2018, Cipullo et al. 112 2018, Basta 2020, Hanley 2020, Scheckel et al. 2019, Menzie et al. 2008). In addition to addressing these 113 data gaps, this study provides an example of combining information across scientific disciplines to yield 114 findings that are important to regulatory decision making. For example, it has been hypothesized that rapidly changing climatic conditions including the thawing of permafrost may affect the stability of 115 116 contaminant and nutrient cycles in the Arctic tundra (Alekseev et al. 2021). However, soil studies from 117 Arctic locations near human populations that rely on subsistence hunting, harvesting, and fishing remain 118 severely under-represented, and available data on metals in Alaskan soil are insufficient to assess 119 exposure in such populations (Perryman et al. 2020, Camenzuli et al. 2014; Cipullo et al. 2018; Yang et 120 al. 2016). This study addresses data gaps by combining information across scientific disciplines to yield 121 findings that are important to regulatory decision making.

## 122 Materials and Methods

A brief description of the sample collection and IVBA extraction methods is provided below. For a more robust discussion of the methods employed herein, the reader is directed to Shock et al (2007).

## 125 Soil Sample Collection

126 Two tundra soil grab samples were collected during June and July 2018 for metals analysis and IVBA

127 testing. Samples were collected from locations previously sampled in 2005 to allow temporal comparison

- 128 with the results reported by Shock et al. (2007). One of the sampling sites was located approximately
- 129 2000 m to 3000 m downwind of the active mine area, where Ba-enriched dust deposition likely occurred.
- 130 This sample is referred to as the "mine sample" or "mine location." The other sampling site was located

- 131 approximately 10 m downwind from the DMTS transport road, where windblown dust from the road and
- 132 truck traffic was deposited (Figure 1). This sample is referred to as the "road sample" or "road location."





Figure 1: Study area with locations of tundra soil samples



#### 144

## Total Metals Determination

145 To remain consistent with the prior data collection methods and to allow comparison between the

146 2005 and 2018 datasets, laboratory X-ray fluorescence analysis (XRF) was used to measure the total Al

- 147 and Ba concentrations in the soil samples.
- 148

#### In Vitro Bioaccessibility Testing

- 149 IVBA testing was conducted following U.S. EPA Method 1340. This protocol involves extracting a 1-g
- aliquot of the test soil in 100 mL of buffered extraction fluid (i.e., simulated gastric fluid) at pH 1.5 for
- 151 one hour with end-over-end rotation. IVBA extraction fluid was analyzed in 2018 using inductively
- 152 coupled plasma mass spectrometry (ICP-MS)<sup>1</sup>. At the end of the extraction process, the mass of metal
- 153 extracted was compared to the total mass of the metal in a separate aliquot of the test material. IVBA was

154 calculated and expressed on a percentage basis using the following equation:

- 155 metal<sub>ext</sub> = in vitro extractable metal in the in vitro extract (mg/L)
- 156 V<sub>ext</sub> = extraction solution volume (L)
- 157 Metal<sub>soil</sub> = metal concentration in the soil sample being assayed (mg/kg)
- 158 Soil<sub>mass</sub> = mass of soil sample being assayed (kg)

# 159 **Results and Discussion**

- 160 Tables 1 and 2 provide a summary of the 2005 and 2018 total metals concentrations and the IVBA
- 161 extraction testing results for the two different soil particle size fractions, for Al and Ba, respectively.

#### 162 Aluminum

- 163 Particle size had no consistent influence on the concentration or bioaccessibility (as measured by
- 164 IVBA) of Al in tundra soils collected from either time period (Table 1)<sup>2</sup>. Al soil concentrations as
- 165 measured in 2018 were lower in both the road and mine samples relative to samples collected from the
- same locations in 2005 (Table 1), suggesting a decrease in Al concentration over time with the greatest

<sup>&</sup>lt;sup>1</sup> This differs from 2005 where the extracts were analyzed using inductively coupled plasma-optical emission spectroscopy (ICP-OES) following a modified version of EPA Method 6010B (Shock et al. 2007).

<sup>&</sup>lt;sup>2</sup> Al concentration was not measured in the <2 mm particle size fraction due to low mass of the 2018 sample

decrease observed for the road location. The bioaccessibility of Al from tundra soils was very low, for
both the 2005 and 2018 samples: with IVBA ranging from 1.3% to 5.5% for the 2018 samples and from
0.5% to 3.3% for the 2005 samples (Table 1). The data from both years indicate that the bioaccessibility
of Al is not strongly dependent on soil particle size, though a small decrease in IVBA was observed with
decreasing particle size for soil samples from both years.

| Tundra Soil Near Road         <2mm Particle Size Fraction         2018 Soil Sample       14000       1.4       0.5         2005 Soil Sample       32100       0.5       0.5         <250µm Particle Size Fraction       2018 Soil Sample       13800       1.3       0.5         2018 Soil Sample       13800       1.3       0.5       0.5         Tundra Soil Near Mine   | Sample Location <sup>a</sup> and Particle Size | Total Al in<br>Soil by XRF <sup>b</sup><br>(mg/kg) | IVBA <sup>c</sup><br>(%) | Bias-Adj<br>IVBA <sup>d</sup><br>(%) |
|---|--|--|--------------------------|--------------------------------------|
| <2mm Particle Size Fraction140001.40.52018 Soil Sample321000.50.5<250µm Particle Size Fraction138001.30.52018 Soil Sample138001.30.52005 Soil Sample323000.50.5Tundra Soil Near Mine22<2mm Particle Size Fraction2018 Soil Sample26800°5.52.22018 Soil Sample26800°5.52.22.22005 Soil Sample26800°5.32.12018 Soil Sample268005.32.12018 Soil Sample268005.32.12018 Soil Sample268005.32.12005 Soil Sample268005.32.1at a for 2005 Samples are from Shock et al. (2007); data for solid:fluid ratio of 1:1002014 metals result by X-ray fluorescence at The Mineral LabBA result calculated using total metals result with hydrofluoric acid digestion at TestAmericBA represents mass extracted divided by total mass in soil sample tested, per EPA Method | Tundra Soil Near Road                          |  |                          |                                      |
| 2018 Soil Sample140001.40.52005 Soil Sample321000.5<250µm Particle Size Fraction  | <2mm Particle Size Fraction                    |  |                          |                                      |
| <250µm Particle Size Fraction<br>2018 Soil Sample 13800 1.3 0.5<br>2005 Soil Sample 32300 0.5 Tundra Soil Near Mine <2mm Particle Size Fraction<br>2018 Soil Sample 26800° 5.5 2.2<br>2005 Soil Sample 32600 3.3 <250µm Particle Size Fraction<br>2018 Soil Sample 26800 5.3 2.1<br>2005 Soil Sample 34000 3.2  center: Data for 2005 samples are from Shock et al. (2007); data for solid:fluid ratio of 1:100 Total metals result by X-ray fluorescence at The Mineral Lab /BA represents mass extracted divided by total mass in soil sample tested, per EPA Method  | 2018 Soil Sample<br>2005 Soil Sample           | 14000<br>32100                                     | 1.4<br>0.5               | 0.5                                  |
| 2018 Soil Sample138001.30.52005 Soil Sample323000.5Tundra Soil Near Mine2018 Soil Near Mine2018 Soil Sample26800°5.52.22018 Soil Sample26800°5.52.22005 Soil Sample26800°5.32.12018 Soil Sample268005.32.12018 Soil Sample268005.32.12005 Soil Sample268003.2***********************************  | <250µm Particle Size Fraction                  |  |                          |                                      |
| Tundra Soil Near Mine         <2mm Particle Size Fraction   | 2018 Soil Sample<br>2005 Soil Sample           | 13800<br>32300                                     | 1.3<br>0.5               | 0.5                                  |
| <2mm Particle Size Fraction   | Tundra Soil Near Mine                          |  |                          |                                      |
| 2018 Soil Sample       26800°       5.5       2.2         2005 Soil Sample       32600       3.3           2018 Soil Sample       26800       5.3       2.1         2018 Soil Sample       26800       3.2       2.1         2005 Soil Sample       34000       3.2       2.1         otes:   | <2mm Particle Size Fraction                    |  |                          |                                      |
| 2005 Soil Sample       32600       3.3         <250µm Particle Size Fraction  | 2018 Soil Sample                               | 26800 <sup>e</sup>                                 | 5.5                      | 2.2                                  |
| <250µm Particle Size Fraction<br>2018 Soil Sample 26800 5.3 2.1<br>2005 Soil Sample 34000 3.2           Detes:           Data for 2005 samples are from Shock et al. (2007); data for solid:fluid ratio of 1:100<br>fotal metals result by X-ray fluorescence at The Mineral Lab           /BA result calculated using total metals result with hydrofluoric acid digestion at TestAmeric<br>/BA represents mass extracted divided by total mass in soil sample tested, per EPA Method  | 2005 Soil Sample                               | 32600  | 3.3                      |                                      |
| 2018 Soil Sample       26800       5.3       2.1         2005 Soil Sample       34000       3.2         otes:   | <250µm Particle Size Fraction                  |  |                          |                                      |
| 2005 Soil Sample 34000 3.2<br>otes:<br>Data for 2005 samples are from Shock et al. (2007); data for solid:fluid ratio of 1:100<br>Total metals result by X-ray fluorescence at The Mineral Lab<br>VBA result calculated using total metals result with hydrofluoric acid digestion at TestAmeric<br>VBA represents mass extracted divided by total mass in soil sample tested, per EPA Method   | 2018 Soil Sample                               | 26800  | 5.3                      | 2.1                                  |
| otes:<br>Data for 2005 samples are from Shock et al. (2007); data for solid:fluid ratio of 1:100<br>Total metals result by X-ray fluorescence at The Mineral Lab<br>/BA result calculated using total metals result with hydrofluoric acid digestion at TestAmeric<br>/BA represents mass extracted divided by total mass in soil sample tested, per EPA Method   |  | 34000  | 5.2                      |                                      |
| Data for 2005 samples are from Shock et al. (2007); data for solid:fluid ratio of 1:100<br>Total metals result by X-ray fluorescence at The Mineral Lab<br>VBA result calculated using total metals result with hydrofluoric acid digestion at TestAmeric<br>VBA represents mass extracted divided by total mass in soil sample tested, per EPA Method  | otes:  |  |                          |                                      |
| Total metals result by X-ray fluorescence at The Mineral Lab<br>VBA result calculated using total metals result with hydrofluoric acid digestion at TestAmeric<br>VBA represents mass extracted divided by total mass in soil sample tested, per EPA Method   | Data for 2005 samples are from Shock e         | t al. (2007); data for s                           | solid:fluid ratio c      | of 1:100                             |
| VBA result calculated using total metals result with hydrofluoric acid digestion at TestAmeric<br>VBA represents mass extracted divided by total mass in soil sample tested, per EPA Method   | Total metals result by X-ray fluorescence      | e at The Mineral Lab                               |                          |                                      |
| VBA represents mass extracted divided by total mass in soil sample tested, per EPA Method   | VBA result calculated using total metals       | result with hydrofluor                             | ic acid digestion        | n at TestAmeric                      |
|   | VBA represents mass extracted divided          | by total mass in soil s                            | sample tested, p         | er EPA Method                        |

174 Under the standard IVBA extraction methods there were "interferences" between components of the extraction fluid and the Al mass in that fluid (ACZ Laboratory personal communication)<sup>3</sup>. These 175 176 interferences result in reported concentrations of Al in the extraction fluid that are biased high. Quality assurance (QA) data from 2018 showed that ICP-MS recovery of Al spiked into the IVBA extraction fluid 177 178 was 152% to 388%, indicating the reported IVBA values are overestimated. Table 1 presents both the 179 original, unadjusted IVBA values and values adjusted by a factor of 2.5 for the high bias (based on the 180 average recovery across triplicate samples, 247%, or a factor of approximately 2.5). For samples collected 181 in 2005 (reported by Shock et al. 2007), bias was not an issue because the IVBA fluid was analyzed using 182 ICP-OES. For any future efforts related to determination of Al IVBA, this potential for bias should be 183 considered in the selection of an analytical lab and the type of instrumentation. When adjusted for the 184 analytical bias, the bioaccessibility of Al from the 2018 soil samples was the same or lower than that 185 measured in samples from matched locations in 2005. 186 Barium 187 There was no consistent effect of soil particle size on either the concentration or bioaccessibility of Ba in soils collected from either year (Table 2)<sup>4</sup>. Ba soil concentrations were similar between 2005 and 188 189 2018: concentrations ranged from 3,000 mg/kg to 6,300 mg/kg in 2018 samples, and from 2,990 mg/kg to

190 8,560 for samples collected in 2005. As was seen with Al, Ba soil concentrations were higher in mine

- samples as compared to those from the road location. As with the results for Al, the reported
- 192 bioaccessibility of Ba was low for soil samples collected from both time periods: with IVBA ranging
- 193 from 11% to 22% for the 2018 samples, and from 12% to 20% for the 2005 samples (Table 2). Ba
- 194 bioaccessibility was greater in road samples collected in 2018 as compared to 2005 samples from the

<sup>&</sup>lt;sup>3</sup> According to the analytical laboratory: "The IVBA extraction and analysis procedure has not been optimized for aluminum. Having off-method analytes out of control limits is not unusual considering the IVBA extraction matrix is made of pH adjusted glycine, which behaves differently in the ICP-MS plasma than in an acidified DI [deionized] water blank. Glycine contains lots of carbon, and carbon enhances the energy in the plasma, causing some elements to be biased high, while other elements will see less bias. This is due to varying elemental ionization potentials. Since the laboratory fortified blank (LFB) and the matrix spike (MS) samples showed high bias for Al, one could assume all results are biased high for those analytes."

<sup>&</sup>lt;sup>4</sup> As with Al, the Ba concentration could not be measured in the <2 mm particle size fraction due to low mass of the 2018 sample.

- same location, but the same trend was not seen for the mine samples. There was no analytical bias for the
- 196 Ba IVBA samples.
- 197
- 198

| and Particle Size                    | Total Al in Soil by XRF <sup>b</sup><br>(mg/kg) | IVBA <sup>c</sup><br>(%) |
|--------------------------------------|---|--------------------------|
| Tundra Soil Near Road                |   |                          |
| <2mm Particle Size Fraction          |   |                          |
| 2018 Soil Sample<br>2005 Soil Sample | 3000<br>3230                                    | 22<br>12                 |
| 250µm Particle Size Fraction         |   |                          |
| 2018 Soil Sample<br>2005 Soil Sample | 3200<br>2990                                    | 21<br>13                 |
| Tundra Soil Near Mine                |   |                          |
| <2mm Particle Size Fraction          |   |                          |
| 2018 Soil Sample                     | 6300 <sup>d</sup>                               | 13                       |
| 2005 Soil Sample                     | 8560  | 13                       |
|                                      |   |                          |
| 250μm Particle Size Fraction         |   |                          |

<sup>b</sup> IVBA result calculated using total metals result with hydrofluoric acid digestion at TestAmerica

<sup>c</sup> NBA represents mass extracted divided by total mass in soil sample tested, per EPA Method 1340

 $^{d}$  Not measured due to low sample mass in <2mm fraction; value here represents results for <250  $\mu m$ 

199

# 200 Effect of Time on Bioaccessibility

201 Although adjustments for bioavailability are sometimes incorporated into ecological and human

202 health risk assessments, few studies have documented the stability of metals bioavailability in soils over

time. Liang et al. (2016) and Xu et al. (2019) conducted short-term studies that evaluated changes over

time in IVBA for soils spiked with soluble lead, arsenic, and cadmium in the laboratory. Bioaccessibility

205 initially decreased, especially in the first month after spiking, and then stabilized after 76 weeks under

controlled laboratory conditions. Studies on chemical amendments added to soils with the specific
intention of reducing the bioavailability of contaminants (e.g., Scheckel et al. 2005 and 2013; Ryan et al.
2004, Scheckel and Ryan 2004) have investigated changes in metals bioaccessibility over time. These
authors specifically discuss the limitations associated with these bench-scale laboratory experiments and
the "general lack of field-scale testing" which make it difficult to translate these results to the natural
environment.

212 A query of researchers and regulators indicates that assessing temporal changes in the bioavailability 213 of chemicals from soils in the natural environment has been largely unstudied (Hanley, personal 214 communication). Academic researchers involved in this field over the last several decades note that short 215 term decreases in bioavailability have been established for soils under laboratory conditions and for tests 216 with chemically amended soils, but data from long-term in situ studies are lacking (Basta, personal 217 communication). Bradham et al. (2018) conducted long-term in situ studies on soils in Joplin, Missouri, where different soil amendments were tested for their ability to reduce the bioavailability of Pb. Their 218 219 research suggests that specific soil amendments effectively reduced Pb bioavailability, which remained 220 stable for 16 years after treatment.

To our knowledge, this study is one of the first to examine the IVBA of Al and Ba from soils in a natural environment over time. Results of this study suggest that for the Arctic tundra soils in the vicinity of Red Dog Mine, the bioavailability of these metals (as approximated by IVBA) has remained generally stable over the more than a decade.

225

#### Bioavailability in Arctic Environments

Few studies have evaluated the bioaccessibility of metals from tundra soils. Brumbaugh et al. (2011) collected vegetation and dust samples along the DMTS haul road as it passes through Cape Krusenstern Nation Monument and evaluated the samples for metals bioaccessibility using the same IVBA methods as this study. They reported low to intermediate (20% to 40%) bioaccessibility of Ba from road dust and vegetation and relatively low (<6%) bioaccessibility of Al. Brumbaugh et al. (2011), the bioaccessibility results are limited to dusts and vegetation (rather than the subsurface peat samples that most closely match the matrix reported here), thus precluding direct comparison across studies. Nevertheless, results
from both studies suggest low bioaccessibility of Al and Ba. It has been hypothesized that rapidly
changing climatic conditions may affect the stability of contaminant and nutrient cycles in the Arctic
tundra (Alekseev et al. 2021, Perryman et al. 2020, Camenzuli et al. 2014, Cipullo et al. 2018, Yang et al.
2016). These studies have found that data on metals concentrations in Arctic soils from areas near
subsistence communities are lacking, and the authors have identified a critical need for such studies in
areas with continuous permafrost.

239 Permafrost-affected soils in the Arctic occupy a significant land area and are notably unique 240 compared to soils from other geographical regions (Alekseev et al. 2021). Research documenting the 241 behavior of metals and metalloids in Artic ecosystems has been labeled as of "great interest" and data that 242 can enhance the environmental management of these areas is of "crucial" social and economic importance 243 (Tregubova et al. 2021). Perryman et al. (2020) cautioned that critical gaps exist for metals data for 244 continuous permafrost zones in Northern Alaska. In a review of remediation technologies for metals-245 contaminated sites in polar regions, Camenzuli et al. (2014) illustrated the unique factors affecting this 246 environment, pointing out that environmental contamination from human activities "remains understudied 247 and unresolved in many polar regions." Complex chemical and biological interactions (e.g., pH, 248 competitive sorption/desorption, precipitation, microbial metabolism, oxidation/reduction) are known to 249 influence the mobility of metals from soils in the natural environment. These factors and the studies 250 mentioned above illustrate the importance and need for in situ studies of the nature and stability of 251 contaminants in Arctic environments. This study on the bioaccessibility of Al and Ba from Arctic tundra 252 soils can help fill such data gaps.

253

#### Effects of Soil Particle Size

Investigations into the soil-chemical interactions that affect the solubility and bioavailability of metals from soil have long indicated that soil particle size can be an important consideration, and that the higher surface area ratio of small particles can sometimes result in greater liberation of metals (Ruby et al. 1999, U.S. EPA 2007a, Karna et al. 2017). Research also indicates that such relationships may also depend on 258 other factors such as site-specific soil characteristics (e.g., Ruby et al. 1999, U.S. EPA 2007a, Li et al. 259 2019, Ma et al. 2019, Ljung et al. 2007, Quin et al. 2016). Such considerations may be particularly 260 important for understanding the potential human health risks from exposures to contaminants in soil 261 because typically, smaller soil particle size more accurately represent the fraction of soil that is 262 incidentally ingested (Ruby and Lowney 2012, U.S. EPA 2016). 263 Our study showed little effect of soil particle size on either Al and Ba concentrations or IVBA. This 264 observation is consistent for samples collected in both 2005 and 2018, based on the <250 µm and <2 mm 265 particle size fractions. The smaller particle size fraction now recommended by U.S. EPA (2016) for use in human health risk assessments, <150 µm, should be considered for future studies examining the effects of 266

soil particle size on IVBA, although the data reported here for Al and Ba suggest that there may be littledifference.

# 269 Summary and Conclusions

The primary objectives for this study were to determine if the concentrations and bioaccessibility of Al and Ba in Arctic tundra soils collected from two locations near Red Dog Mine changed over time, and to assess whether soil particle size had any influence over these metrics. The data from this study show:

- Al tundra soil concentrations were lower in 2018 as compared to in 2005, while Ba
   concentrations generally remained the same over time.
- The bioaccessibility of Al and Ba from tundra soils collected in the vicinity of Red Dog Mine and
   its transportation corridor is low (maximum 5.5% unadjusted IVBA for Al, and maximum 22%
   IVBA for Ba).
- A small decrease in Al bioaccessibility was observed with decreasing particle size for soil
   samples from both years, but soil particle size did not predictably affect the concentrations or
   bioaccessibility of Al or Ba.

| 281 | • | While there were slight differences in the measured bioaccessibility of Al from the two time        |
|-----|---|---|
| 282 |   | points, the IVBA values are all very low, suggesting little to no change in the bioaccessibility of |
| 283 |   | Al from the tundra soils over time.   |

Ba bioaccessibility was greater in road samples collected in 2018 as compared to 2005 samples
 from the same location.

• Analytical bias affected the IVBA results for Al but not Ba; potential for bias should be considered when selecting an analytical lab and the type of instrumentation in future studies.

288 This investigation of whether Al and Ba tundra soil concentrations and bioaccessibility changed over 289 time or were related to soil particle size was conducted as part of the ongoing RDO fugitive dust risk 290 management program. Uncertainty reduction studies combined with monitoring results such as these are 291 used by RDO to verify the effectiveness of fugitive dust best management practices and operational 292 improvements, and to inform nearby communities that utilize the areas surrounding RDO for subsistence 293 hunting and gathering. The IVBA data reported in Shock et al. (2007) and the data from this study 294 indicate that the bioavailability of Al and Ba from tundra soils is likely to be substantially lower than the 295 default assumption of 100% bioavailability used in the fugitive dust risk assessment. This illustrates the 296 importance for incorporating bioavailability adjustments for risk assessment of Arctic soils. 297 This study provides information on the concentrations and bioaccessibility of Al and Ba in Arctic

tundra soils, a topic that multiple researchers, regulators, health scientists, and soil scientists have
identified as a data gap. Another unique aspect of this study is that it provides information about the insitu stability of the concentrations and IVBA of the two metals in Arctic soils over time. The
bioavailability of Al and Ba from tundra soils, as approximated by IVBA, is relatively low and has not
changed substantially since 2005. The information provided in this study along with the study by Shock et
al. (2007) improves the understanding of Al and Ba bioaccessibility from soils and can be used to refine
the results of human health and ecological risk assessments at mining sites. In addition, considerations

- 305 regarding analytical methods and particle size effects, along with information that is specific to Arctic
- 306 tundra soils may be used to inform future investigations.

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- 467 10.1016/j.envpol.2016.04.069. PMID: 27131808.

| 7. | Appendix A | General comment | EPA has validated an IVBA assay for predicting soil arsenic and lead RBA for use in human health risk assessments, but has not done so for the other metals mentioned. The DEC risk assessment manual adopted in regulation clearly states that a default value of 100% is recommended for all chemicals except arsenic and lead. EPA has not validated IVBA assays for other metals in respect to human health. However, the information can be presented in the uncertainty section in a human health risk assessment and in applications for lead and arsenic is discussed but it should be made clear that the application regarding quantitative risk assessment with the other metals is suggesting meeting 100% of the requirements in 18 AAC 75 without a validated method. |  |
|----|------------|-----------------|---|--|
|    |            |                 | vandated method.  |  |

Understood and agreed. EPA has validated the IVBA method only for As and Pb for quantitative use in risk assessment, as was stated in third paragraph of "Study Background" section. However, for the purposes of this study, the IVBA method was applied to Al and Ba as a tool to understand more about the behavior of these metals in the environment, and most recently to assess whether there have been changes over time in the stability of the metals in the arctic environment, not for any application to risk assessment. This is discussed in the Study Background:

This method has been validated for quantitative use in predicting relative oral bioavailability of Pb and arsenic (As) for human health risk assessment (U.S. EPA 2017). Other investigations have also established that the acidic extraction conditions used in EPA Method 1340 also generate data that are generally predictive of the bioavailability of cadmium (Cd), for which an International Organization for Standardization (ISO) Standard has been developed (ISO 2018; Schroder et al. 2003; Boros et al. 2017). Similarly, investigations by Health Canada indicate that a low pH extraction method, such as EPA Method 1340, results in the most conservative estimate of IVBA for zinc (Zn) (Koch et al. 2013). Therefore, while validated by U.S. EPA (2018) for Pb and As only, EPA Method 1340 can also provide screening information for the bioavailability of other metals (Menzie et al. 2008).

| 8. | Pg 40-01 | Tables 1 and 2 | The total metal concentration in the soil was performed by XRF, a screening value/ tool dependent upon correlation and calibration with the respective soil media. However, this estimated value was used to determine IVBA with a different analytical analysis for the extract. The extract was based off ICP-AES (2005) and ICP-AES (2018). The same laboratory approved method for the "soil and extract" from EPA metal analysis 6010D or 6020B or 7000 series as noted in DEC Field Sampling Guidance Oct. 2019 is recommended. |
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Thank you for the comment. Please note, the primary goal of the current IVBA evaluation of Al and Ba was specifically intended to understand changes over time. The initial effort, reported in Shock et al. (2007), used laboratory XRF results to calculate IVBA for these metals. Therefore, for this comparison to be meaningful, it was appropriate to base the IVBA calculations on total metal determination with XRF.

EPA Method 3050B for soil <u>digestion</u> may not recover (dissolve) most of the Al and Ba in Red Dog site soils. As reported by Shock et al. (2007), comparison of results from this standard EPA digestion method for assessing total metals in soil against data from XRF evaluation identified that the EPA method extracted between 0.1% and 100% of Ba from site samples and 10% to 32% of Al from site samples. This is irrespective of the analytical instrumentation used (e.g., EPA 6010D, 6020B or 7000 series). This was an important finding by Shock et al. (2007), who concluded the following:

many usur considerations for Louinaung Diouvanaomity. This study illustrates that the type of analyses used to assess barium concentrations in sampled substrates can have significant impact on calculated bioaccessibility. The extractable barium (Method 3050B/ICP-OES) was only a minor fraction of the total (XRF) barium, and therefore underestimated total concentrations in the main waste road and gyro crusher dust samples, and to a lesser extent, in the tundra soil samples. This occurs because barium in the form of barite is recalcitrant to the standard soil digestion methods (i.e., acid digestion by EPA Method 3050B), whereas XRF analysis reports higher barium concentrations for these soil media (effectively reporting total concentrations). If barium bioaccessibility is calculated using concentrations measured from standard digestion/analysis methods, the resulting values will likely be higher than would be obtained using XRF analysis.

Said another way, soils and IVBA fluids are often evaluated with different instrumentation. Conventionally, IVBA for metals in soil involves assessment of total metals, often extraction/digestion EPA Methods 3050 or 3051, and IVBA with extraction methods specified in EPA Method 1340. Under EPA Method 3050 (total metals in sediment sludges and soils), ICP-AES is recommended. However, the method also states that "alternative determinative techniques may be used." Shock et al. (2007) provided a comparison of total concentrations of Al and Ba in soils from the Red Dog area based on analysis with EPA Method 3050 (with ICP-OES) and XRF. Their results demonstrated that EPA Method 3050 does not capture the total concentrations of these target metals present [Note, EPA Method 3050 specifically states that it is a strong acid digestion but "is not a total digestion technique for most samples"]. For example, Shock et al. reported that concentrations of Ba in the <250 particle size fraction for tundra soils via EPA Method 3050 ranged from 58% to 97% of the concentration reported by laboratory XAS (for other samples, Method 3050 recovered only <1% of total Ba in the sample), and suggests that results are due to the presence of insoluble barite, which is not solubilized by the digestion specified in EPA Method 3050. Similarly, for Al, Shock et al. report that concentrations determined via Method 3050 were 16% to 24% of those reported via XRF, a result of incomplete digestion of silicate minerals that would contain Al. Therefore, Shock et al. selected the XRF method for reporting total metal concentrations, and these were used as the basis for calculating bioaccessible Ba and Al.

For this evaluation of IVBA, XRF was used to assess total concentrations of Al and Ba to circumvent the potential incomplete extraction associated with EPA Method 3050B, and to generate results that are comparable against those reported by Shock et al. (2007) given that the primary goal of the effort was to allow comparison of samples from two different time points.

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The large bias range (e.g. Al = 152% to 388%) suggests it would be appropriate to include the standard deviation with the bias.

The values for range in bias was provided for discussion of implications from the QA/QC data. In this case, the bias is indicated by IVBA spiked with soluble aluminum, and the recovery values are 152% 199%, and 388% of the spiked concentration. The first two values represent IVBA extraction of a spiked soil, and the 388% value represents recovery of Al from a spiked IVBA fluid. Given the limited number of samples and that all values are disclosed, we did not provide the standard deviation of the bias. For clarity, we rearranged the text as follows:

Under the standard IVBA extraction methods there were "interferences" between components of the extraction fluid and the Al mass in that fluid (ACZ Laboratory personal communication). These interferences result in reported concentrations of Al in the extraction fluid that are biased high. Quality assurance (QA) data from 2018 showed that ICP-MS recovery of Al spiked into the IVBA extraction fluid was 152% to 388%, indicating the reported IVBA values are overestimated. Table 1 presents both the original, unadjusted IVBA values and values adjusted by a factor of 2.5 for the high bias (based on the average recovery across triplicate samples, 247%, or a factor of approximately 2.5). For samples collected in 2005 (reported by Shock et al., 2007), bias was not an issue because the IVBA fluid was analyzed using ICP-OES. For any future efforts related to determination of Al IVBA, this potential for bias should be considered in the selection of an analytical lab and the type of instrumentation. When adjusted for the analytical bias, the bioaccessibility of Al from the 2018 soil samples was the same or lower than that measured in samples from matched locations in 2005.

| Ē. |  | <ul> <li>Some of the details within the body of the tables are unclear.</li> <li>1) Sample size should be presented (n=XX)</li> <li>2) Since sample size is not presented it is unclear if "%" is based upon an average or a single sample (please note for clarity). DEC regulation is based on maximum or approved 95% upper average concentrations to soil.</li> </ul> |
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EPA Method 1340 for assessing the IVBA of Pb and As in soils specifies QA/QC measures that include blanks, spikes, and replicates, but does not indicate the need for replicate extraction of all soil samples. The data presented represent individual samples from each location. Replicate samples included in the extraction process indicated good reproducibility of extraction results for either soil particle size fraction. Consistent with EPA Method 1340 for IVBA analysis, individual data points presented do not represent averages of replicates.

All soil concentrations that were previously incorporated into the 2007 risk assessment process represent values consistent with DEC regulation.

|  | 3) The raw data that were used in creating the table should be provided,<br>and either cited if from prior reports or the actual data provided in a<br>separate attachment. |  |
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Please find an attachment with the raw data that were used for creating the table.

|                                  |               |                      |   |                                  |  |   |  |  | Bioaccessibility (%)                 |  |                                     |   |                              |
|----------------------------------|---------------|----------------------|---|----------------------------------|--|---|--|--|--------------------------------------|--|-------------------------------------|---|------------------------------|
| Sample Identifier                | Grain<br>Size | Solid:Fluid<br>Ratio | Total<br>Conc. In<br>Soil <sup>a</sup><br>(mg/kg) | Mass of<br>Soil<br>Tested<br>(g) | Mass of<br>Analyte in<br>Soil<br>Extracted<br>(mg) | Conc. of<br>Analyte in<br>Extract<br>(mg/L) | Mass of<br>Analyte in<br>Extract<br>(mg) | Total Conc.<br>in Soil by<br>XRF <sup>b</sup><br>(mg/kg) | IVBA using<br>Total by<br>XRF<br>(%) | Bias-Adj<br>IVBA using<br>Total by<br>XRF<br>(%) | IVBA using<br>Total by 3050b<br>(%) | Bias-Adj<br>IVBA using<br>Total by 3050b<br>(%) | Ratio of IVBA<br><2mm/<250μm |
| <2mm Particle Size Fraction      |               |                      |   |                                  |  |   |  |  |                                      |  |                                     |   |                              |
| TS-REF-3-COMPOSITE               | <2 mm         | 1:100                | 11900   | 0.999                            | 11.89  | 8.4   | 0.84                                     | 46400  | 1.8                                  | 0.7  | 7.1                                 | 2.8   | 1.1                          |
| TS-REF-7/TS-REF-11 COMPOSITE     | <2 mm         | 1:100                | 2870  | 1.001                            | 2.87   | 7.5   | 0.75                                     |  |                                      |  | 26.1                                | 10.4  | 0.8                          |
| TT3-0010 COMPOSITE               | <2 mm         | 1:100                | 2920  | 1.002                            | 2.93   | 1.9   | 0.19                                     | 14000  | 1.4                                  | 0.5  | 6.5                                 | 2.6   | 1.2                          |
| TT3-0100 COMPOSITE               | <2 mm         | 1:100                | 1490  | 1.000                            | 1.49   | 2.0   | 0.20                                     |  |                                      |  | 13.4                                | 5.4   | 1.1                          |
| TT3-1000 COMPOSITE               | <2 mm         | 1:100                | 809   | 1.000                            | 0.81   | 3.3   | 0.33                                     |  |                                      |  | 40.8                                | 16.3  | 0.9                          |
| TT5-0010 COMPOSITE               | <2 mm         | 1:100                | 7780  | 1.003                            | 7.80   | 3.6   | 0.36                                     | 38600  | 0.9                                  | 0.4  | 4.6                                 | 1.8   | 1.1                          |
| TT5-2000 COMPOSITE               | <2 mm         | 1:100                | 1020  | 0.997                            | 1.02   | 7.0   | 0.70                                     |  |                                      |  | 68.8                                | 27.5  | 1.0                          |
| TT7-0010 COMPOSITE               | <2 mm         | 1:100                | 5740  | 1.002                            | 5.75   | 14.8  | 1.48                                     | 26800  | 5.5                                  | 2.2  | 25.7                                | 10.3  | 1.1                          |
| TT8-0010 COMPOSITE               | <2 mm         | 1:100                | 5600  | 1.000                            | 5.60   | 2.7   | 0.27                                     | 26200  | 1.0                                  | 0.4  | 4.8                                 | 1.9   | 1.1                          |
| TT8-0100 COMPOSITE               | <2 mm         | 1:100                | 3070  | 1.003                            | 3.08   | 3.2   | 0.32                                     | 13500  | 2.4                                  | 0.9  | 10.4                                | 4.2   | 1.0                          |
| TT8-0800/TT8-0900-TT8-1000 COM   | <2 mm         | 1:100                | 726   | 0.999                            | 0.73   | 3.1   | 0.31                                     |  |                                      |  | 42.7                                | 17.1  | 0.9                          |
| 18-289 (Y Road Surface)          | <2 mm         | 1:100                | 12800   | 1.003                            | 12.84  | 2.8   | 0.28                                     | 65600  | 0.4                                  | 0.2  | 2.2                                 | 0.9   | 0.7                          |
| 18-290 (R-Boundary Road Surface) | <2 mm         | 1:100                | 2050  | 0.999                            | 2.05   | 0.7   | 0.07                                     | 9600   | 0.7                                  | 0.3  | 0.0                                 | 0.0   | 0.0                          |
| 18-291 (MS-2 Road Surface)       | <2 mm         | 1:100                | 7040  | 1.000                            | 7.04   | 1.6   | 0.16                                     | 38700  | 0.4                                  | 0.2  | 2.3                                 | 0.9   | 0.8                          |
| 18-292 (Zinc concentrate)        | <2 mm         | 1:100                | 80  | 1.000                            | 0.08   | 0.3   | 0.03                                     | 500  | 6.0                                  | 2.4  | 40.0                                | 16.0  |                              |
| 18-293 (Lead concentrate)        | <2 mm         | 1:100                | 138   | 1.002                            | 0.14   | 0.3   | 0.03                                     | 850  | 3.5                                  | 1.4  | 20.0                                | 8.0   |                              |
| · · ·                            |               |                      | 0   |                                  |  |   |  |  |                                      |  |                                     |   |                              |
| <250µm Particle Size Fraction    |               |                      | 0   |                                  |  |   |  |  |                                      |  |                                     |   |                              |
| TS-REF-3-COMPOSITE               | <250 µm       | 1:100                | 11600   | 1.002                            | 11.62  | 7.41  | 0.741                                    | 46900  | 1.6                                  | 0.6  | 6.4                                 | 2.6   |                              |
| TS-REF-7/TS-REF-11 COMPOSITE     | <250 µm       | 1:100                | 2350  | 0.999                            | 2.35   | 7.72  | 0.772                                    |  |                                      |  | 32.9                                | 13.2  |                              |
| TT3-0010 COMPOSITE               | <250 µm       | 1:100                | 3170  | 1.001                            | 3.17   | 1.78  | 0.178                                    | 13800  | 1.3                                  | 0.5  | 5.6                                 | 2.2   |                              |
| TT3-0100 COMPOSITE               | <250 µm       | 1:100                | 1740  | 0.998                            | 1.74   | 2.16  | 0.216                                    |  |                                      |  | 12.4                                | 5.0   |                              |
| TT3-1000 COMPOSITE               | <250 µm       | 1:100                | 768   | 0.999                            | 0.77   | 3.47  | 0.347                                    |  |                                      |  | 45.2                                | 18.1  |                              |
| TT5-0010 COMPOSITE               | <250 µm       | 1:100                | 7580  | 0.999                            | 7.57   | 3.30  | 0.330                                    | 43400  | 0.8                                  | 0.3  | 4.4                                 | 1.7   |                              |
| TT5-2000 COMPOSITE               | <250 µm       | 1:100                | 1020  | 1.000                            | 1.02   | 7.13  | 0.713                                    |  |                                      |  | 69.9                                | 28.0  |                              |
| TT7-0010 COMPOSITE               | <250 µm       | 1:100                | 6000  | 1.003                            | 6.02   | 14.3  | 1.43                                     | 26800  | 5.3                                  | 2.1  | 23.8                                | 9.5   |                              |
| TT8-0010 COMPOSITE               | <250 µm       | 1:100                | 5260  | 1.004                            | 5.28   | 2.39  | 0.239                                    | 26400  | 0.9                                  | 0.4  | 4.5                                 | 1.8   |                              |
| TT8-0100 COMPOSITE               | <250 µm       | 1:100                | 3350  | 1.000                            | 3.35   | 3.40  | 0.340                                    | 16100  | 2.1                                  | 0.8  | 10.1                                | 4.1   |                              |
| TT8-0800/TT8-0900-TT8-1000 COM   | <250 µm       | 1:100                | 911   | 0.997                            | 0.91   | 4.11  | 0.411                                    |  |                                      |  | 45.3                                | 18.1  |                              |
| 18-289 (Y Road Surface)          | <250 µm       | 1:100                | 13400   | 0.998                            | 13.37  | 4.28  | 0.428                                    | 58700  | 0.7                                  | 0.3  | 3.2                                 | 1.3   |                              |
| 18-290 (R-Boundary Road Surface) | <250 µm       | 1:100                | 2960  | 0.998                            | 2.95   | 1.65  | 0.165                                    | 20400  | 0.8                                  | 0.3  | 5.6                                 | 2.2   |                              |
| 18-291 (MS-2 Road Surface)       | <250 µm       | 1:100                | 9430  | 0.998                            | 9.41   | 2.81  | 0.281                                    | 46500  | 0.6                                  | 0.2  | 3.0                                 | 1.2   |                              |
| 18-292 (Zinc concentrate)        | <250 µm       | 1:100                | 79  | 1.001                            | 0.08   | U   | U  | 500  |                                      |  |                                     |   |                              |
| 18-293 (Lead concentrate)        | <250 µm       | 1:100                | 168   | 1.001                            | 0.17   | U   | U  | 900  |                                      |  |                                     |   |                              |

Note:

<sup>a</sup> Total concentration by 3050b analysis conducted by ACZ Laboratories

<sup>b</sup> IVBA result calculated using total metals result by x-ray fluorescence at The Mineral Lab. --: Not Reported

# Table 2: Aluminum QA sample results from in vitro aluminum bioaccessibility testing of 2018 soil samples

|         |               |       |                             |            |           |        |          |       | Percent  |            |               | Percent          |
|---------|---------------|-------|-----------------------------|------------|-----------|--------|----------|-------|----------|------------|---------------|------------------|
| Grain   |               |       |                             | Extraction | QC        | Sample | Measured |       | Recovery | Acceptable |               | Recovery Recalc  |
| Size    | ACZ ID        | Туре  | Description                 | Date       | Conc.     | Conc.  | Conc.    | Units | (%)      | Range      | Comments      | (%) <sup>a</sup> |
| IVBA EX | TRACTION      |       |                             |            |           |        |          |       |          |            |               |                  |
| <2mm    | WG470898ICB   | ICB   | Blank                       | 4/24/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470898CCB1  | CCB   | Blank                       | 4/24/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470898CCB2  | CCB   | Blank                       | 4/24/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470898CCB3  | CCB   | Blank                       | 4/24/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470417LFB   | LFB   | Spiked IVBA Fluid           | 4/24/2019  | 1.0013    |        | 3.89     | mg/L  | 388      | 80-120%    | Outside range | 388              |
| <2mm    | L51008-03MS   | MS    | Spiked Soil (L51008-03)     | 4/24/2019  | 1.0013    | 1.9    | 4.42     | mg/L  | 252      | 75-125%    | Outside range | 152              |
| <2mm    | L51008-13MS   | MS    | Spiked Soil (L51008-13)     | 4/24/2019  | 1.0013    | 0.7    | 3.38     | mg/L  | 268      | 75-125%    | Outside range | 199              |
| <2mm    | L51008-01DUP  | DUP   | Sample Duplicate            | 4/24/2019  |           | 8.4    | 8.51     | mg/L  | 101      | 80-120%    | Ū.            |                  |
| <2mm    | L51008-12DUP  | DUP   | Sample Duplicate            | 4/24/2019  |           | 2.8    | 2.8      | mg/L  | 100      | 80-120%    |               |                  |
| <2mm    | L51008-06SDL  | SDL   | Serial Dilution             | 4/24/2019  |           | 3.6    | 3.8      | mg/L  | 106      | 90-110%    |               |                  |
| <2mm    | WG470417PBS   | PBS   | Soil Blank                  | 4/24/2019  |           |        | U        | mg/L  |          | <0.3       |               |                  |
| <2mm    | WG470898ICV   | ICV   | Standard                    | 4/24/2019  | 0.1       |        | 0.0965   | mg/L  | 97       | 90-110%    |               |                  |
| <2mm    | WG470898PQV   | PQV   | Standard                    | 4/24/2019  | 0.0150195 |        | 0.0141   | mg/L  | 94       | 70-130%    |               |                  |
| <2mm    | WG470898ICSAB | ICSAB | Standard                    | 4/24/2019  | 50.170026 |        | 46.8947  | mg/L  | 93       | 80-120%    |               |                  |
| <2mm    | WG470898CCV1  | CCV   | Standard                    | 4/24/2019  | 0.50065   |        | 0.4957   | mg/L  | 99       | 90-110%    |               |                  |
| <2mm    | WG470898CCV2  | CCV   | Standard                    | 4/24/2019  | 0.50065   |        | 0.4919   | mg/L  | 98       | 90-110%    |               |                  |
| <2mm    | WG470898CCV3  | CCV   | Standard                    | 4/24/2019  | 0.50065   |        | 0.5049   | mg/L  | 101      | 90-110%    |               |                  |
| TOTAL N | IETAL (3050)  |       |                             |            |           |        |          |       |          |            |               |                  |
| <2mm    | WG470618ICB   | ICB   | Blank                       | 4/19/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470618CCB1  | CCB   | Blank                       | 4/19/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470618CCB2  | CCB   | Blank                       | 4/19/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470618CCB3  | CCB   | Blank                       | 4/19/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470959ICB   | ICB   | Blank                       | 4/25/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470959CCB1  | CCB   | Blank                       | 4/25/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470959CCB2  | CCB   | Blank                       | 4/25/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | WG470959CCB3  | CCB   | Blank                       | 4/25/2019  |           |        | U        | mg/L  |          | <0.015     |               |                  |
| <2mm    | L51008-16MS   | MS    | Spiked Soil (L51008-16)     | 4/19/2019  | 25.0325   | 138    | 176.1    | mg/Kg | 152      | 75-125%    | Outside range | 108              |
| <2mm    | L51008-16MSD  | MSD   | Spiked Soil (L51008-16) Dup | 4/19/2019  | 25.0325   | 138    | 178.3    | mg/Kg | 161      | 75-125%    | Outside range | 109              |
| <2mm    | L51008-16MS   | MS    | Matrix Spike (clean soil)   | 4/25/2019  | 5.0065    | U      | U        | mg/Kg | 0        | 75-125%    | Outside range |                  |
| <2mm    | L51008-16MSD  | MSD   | Matrix Spike (clean soil)   | 4/25/2019  | 5.0065    | U      | U        | mg/Kg | 0        | 75-125%    | Outside range |                  |
| <2mm    | L51008-04SDL  | SDL   | Serial Dilution             | 4/19/2019  |           | 1460   | 1422     | mg/Kg | 97       | 90-110%    |               |                  |
| <2mm    | L51008-03SDL  | SDL   | Serial Dilution             | 4/25/2019  |           | 2920   | 2932.5   | mg/Kg | 100      | 90-110%    |               |                  |
| <2mm    | WG470471PBS   | PBS   | Soil blank                  | 4/19/2019  |           |        | U        | mg/Kg |          | <9         |               |                  |
| <2mm    | WG470471PBS   | PBS   | Soil blank                  | 4/25/2019  |           |        | U        | mg/Kg |          | <9         |               |                  |
| <2mm    | WG470471LCSS  | LCSS  | Soil Standard               | 4/19/2019  | 8360      |        | 8366     | mg/Kg | 100      | 4160-12600 |               |                  |
| <2mm    | WG470471LCSSD | LCSSD | Soil Standard               | 4/19/2019  | 8360      |        | 8661     | mg/Kg | 104      | 4160-12600 |               |                  |
| <2mm    | WG470471LCSS  | LCSS  | Soil Standard               | 4/25/2019  | 8360      |        | 8487     | mg/Kg | 102      | 4160-12600 |               |                  |
| <2mm    | WG470471LCSSD | LCSSD | Soil Standard               | 4/25/2019  | 8360      |        | 8466     | mg/Kg | 101      | 4160-12600 |               |                  |
| <2mm    | WG470618ICV   | ICV   | Standard                    | 4/19/2019  | 0.1       |        | 0.0957   | mg/L  | 96       | 90-110%    |               |                  |
| <2mm    | WG470618PQV   | PQV   | Standard                    | 4/19/2019  | 0.0150195 |        | 0.0144   | mg/L  | 96       | 70-130%    |               |                  |
| <2mm    | WG470618ICSAB | ICSAB | Standard                    | 4/19/2019  | 50.170026 |        | 47.0836  | mg/L  | 94       | 80-120%    |               |                  |
| <2mm    | WG470618CCV1  | CCV   | Standard                    | 4/19/2019  | 0.50065   |        | 0.4963   | mg/L  | 99       | 90-110%    |               |                  |

# Table 2: Aluminum QA sample results from in vitro aluminum bioaccessibility testing of 2018 soil samples

|         |                |       |                              |            |           |        |          |               | Percent  |                   |               | Percent          |
|---------|----------------|-------|------------------------------|------------|-----------|--------|----------|---------------|----------|-------------------|---------------|------------------|
| Grain   |                |       |                              | Extraction | QC        | Sample | Measured |               | Recovery | Acceptable        |               | Recovery Recalc  |
| Size    | ACZ ID         | Туре  | Description                  | Date       | Conc.     | Conc.  | Conc.    | Units         | (%)      | Range             | Comments      | (%) <sup>a</sup> |
| <2mm    | WG470618CCV2   | CCV   | Standard                     | 4/19/2019  | 0.50065   |        | 0.5064   | mg/L          | 101      | 90-110%           |               | · ·              |
| <2mm    | WG470618CCV3   | CCV   | Standard                     | 4/19/2019  | 0.50065   |        | 0.524    | mg/L          | 105      | 90-110%           |               |                  |
| <2mm    | WG470959ICV    | ICV   | Standard                     | 4/25/2019  | 0.1       |        | 0.0962   | mg/L          | 96       | 90-110%           |               |                  |
| <2mm    | WG470959PQV    | PQV   | Standard                     | 4/25/2019  | 0.0150195 |        | 0.0124   | mg/L          | 83       | 70-130%           |               |                  |
| <2mm    | WG470959ICSAB  | ICSAB | Standard                     | 4/25/2019  | 50.170026 |        | 47.325   | mg/L          | 94       | 80-120%           |               |                  |
| <2mm    | WG470959CCV1   | CCV   | Standard                     | 4/25/2019  | 0.50065   |        | 0.4999   | mg/L          | 100      | 90-110%           |               |                  |
| <2mm    | WG470959CCV2   | CCV   | Standard                     | 4/25/2019  | 0.50065   |        | 0.5114   | mg/L          | 102      | 90-110%           |               |                  |
| <2mm    | WG470959CCV3   | CCV   | Standard                     | 4/25/2019  | 0.50065   |        | 0.4976   | mg/L          | 99       | 90-110%           |               |                  |
| IVBA EX | TRACTION       |       |                              |            |           |        |          |               |          |                   |               |                  |
| <250um  | WG471151ICB    | ICB   | Blank                        | 4/29/2019  |           |        | U        | mg/L          |          | <0.015            |               |                  |
| <250um  | WG471151CCB1   | CCB   | Blank                        | 4/29/2019  |           |        | U        | mg/L          |          | <0.015            |               |                  |
| <250um  | WG471151CCB2   | CCB   | Blank                        | 4/29/2019  |           |        | U        | mg/L          |          | <0.015            |               |                  |
| <250um  | WG471151CCB3   | CCB   | Blank                        | 4/29/2019  |           |        | U        | mg/L          |          | <0.015            |               |                  |
| <250um  | WG470840LFB    | LFB   | Spiked IVBA Fluid            | 4/29/2019  | 1.0013    |        | 2.668    | mg/L          | 266      | 80-120%           | Outside range | 266              |
| <250um  | L51009-03MS    | MS    | Spiked Soil (L51009-03)      | 4/29/2019  | 1.0013    | 1.78   | 4.283    | mg/L          | 250      | 75-125%           | Outside range | 154              |
| <250um  | L51009-16MS    | MS    | Spiked Soil (L51009-16)      | 4/29/2019  | 1.0013    | U      | 3.4      | mg/L          | 340      | 75-125%           | Outside range | 340              |
| <250um  | L51009-01DUP   | DUP   | Sample Duplicate             | 4/29/2019  |           | 7.41   | 7.517    | mg/L          | 101      | 20                |               |                  |
| <250um  | L51009-15DUP   | DUP   | Sample Duplicate             | 4/29/2019  |           | U      | U        | mg/L          |          | 80-120%           |               |                  |
| <250um  | L51009-06SDL   | SDL   | Serial Dilution              | 4/29/2019  |           | 3.3    | 3.715    | mg/L          | 113      | 80-120%           |               |                  |
| <250um  | WG470840PBS    | PBS   | Soil Blank                   | 4/29/2019  | <b>.</b>  |        | U        | mg/L          |          | <0.09             |               |                  |
| <250um  | WG471151ICV    | ICV   | Standard                     | 4/29/2019  | 0.1       |        | 0.0933   | mg/L          | 93       | 90-110%           |               |                  |
| <250um  | WG4/1151PQV    | PQV   | Standard                     | 4/29/2019  | 0.0150195 |        | 0.0139   | mg/L          | 93       | 70-130%           |               |                  |
| <250um  | WG471151ICSAB  | ICSAB | Standard                     | 4/29/2019  | 50.170026 |        | 48.3641  | mg/L          | 96       | 80-120%           |               |                  |
| <250um  | WG4/1151CCV1   |       | Standard                     | 4/29/2019  | 0.50065   |        | 0.5025   | mg/L          | 100      | 90-110%           |               |                  |
| <250um  | WG471151CCV2   |       | Standard                     | 4/29/2019  | 0.50065   |        | 0.5054   | mg/L          | 101      | 90-110%           |               |                  |
|         |                |       | Standard                     | 4/29/2019  | 0.50065   |        | 0.5082   | mg/L          | 102      | 90-110%           |               |                  |
|         | IE I AL (3050) | 100   |                              | 4/05/0040  |           |        |          |               |          | .0.045            |               |                  |
| <250um  | WG4/1030ICB    | ICB   | Blank                        | 4/25/2019  |           |        | U        | mg/L          |          | < 0.015           |               |                  |
| <250um  | WG4/1030CCB1   | CCB   | Blank                        | 4/25/2019  |           |        | U        | mg/L          |          | < 0.015           |               |                  |
| <250um  | WG471030CCB2   | CCB   | Blank                        | 4/25/2019  |           |        | 0        | mg/L          |          | < 0.015           |               |                  |
| <250um  | WG4/1030CCB3   | CCB   | Blank                        | 4/25/2019  |           |        | U        | mg/L          |          | < 0.015           |               |                  |
| <250um  | WG471219ICD    |       | Dialik                       | 4/29/2019  |           |        | 0        | mg/∟<br>mg/l  |          | <0.015            |               |                  |
| <250um  | WG471219CCB1   |       | Dialik                       | 4/29/2019  |           |        | 0        | mg/∟<br>mg/l  |          | <0.015            |               |                  |
| <250um  | WG47121900B2   |       | Dialik<br>spikod 3050 fluid  | 4/29/2019  | 0.050065  |        | 0.0476   | mg/L<br>mg/Kg | 05       | <0.015<br>80.120% |               |                  |
| <250um  | WG470762LFD2   |       | spiked 3050 fluid            | 4/25/2019  | 0.050005  |        | 0.0470   | mg/Kg         | 90       | 80 120%           |               |                  |
| <250um  | 151000_00MS    | MS    | Spiked Soil (1 51009-09)     | 4/25/2019  | 25 0325   | 5510   | 5842.8   | mg/Kg         | 1320     | 75-125%           | Outside range | 106              |
| <250um  |                | MSD   | Spiked Soil (151009-09)      | 4/25/2019  | 25.0325   | 5510   | 5663 5   | mg/Kg         | 613      | 75-125%           | Outside range | 102              |
| <250um  | 1 51009-09MSD  | MS    | Spiked Soil (151009-09) Dup  | 4/29/2019  | 25.0525   | 5260   | 5648     | ma/Ka         | 1552     | 75-125%           | Outside range | 102              |
| <250um  | 1 51009-09MSD  | MSD   | Spiked Soil (1 51009-09) Dup | 4/29/2019  | 25        | 5260   | 5609     | ma/Ka         | 1396     | 75-125%           | Outside range | 106              |
| <250um  | 1 51009-03SDI  | SDI   | Serial Dilution              | 4/25/2019  | 20        | 3170   | 2971     | ma/Ka         | 94       | 90-110%           | Satolas range | 100              |
| <250um  | L51009-11SDI   | SDL   | Serial Dilution              | 4/29/2019  |           | 1040   | 1037     | ma/Ka         | 100      | 90-110%           |               |                  |
| <250um  | WG470762PBS    | PBS   | Soil blank                   | 4/25/2019  |           |        | U        | mg/Kg         |          | <9                |               |                  |

# Table 2: Aluminum QA sample results from in vitro aluminum bioaccessibility testing of 2018 soil samples

| Grain  |               |       |               | Extraction | 00        | Samplo | Mossured |        | Percent | Accontable |               | Percent<br>Receivery Receiver |
|--------|---------------|-------|---------------|------------|-----------|--------|----------|--------|---------|------------|---------------|-------------------------------|
| Sizo   |               | Tuno  | Description   | Data       | Cono      | Cono   | Cono     | Llaita | (04)    | Banga      | Commonto      |                               |
| Size   | ACZ ID        | туре  | Description   | Dale       | COLC.     | CONC.  | CONC.    | Units  | (70)    | Range      | Comments      | (%)                           |
| <250um | WG470843PBS   | PBS   | Soil blank    | 4/25/2019  |           |        | U        | mg/Kg  |         | <9         |               |                               |
| <250um | WG470843PBS   | PBS   | Soil blank    | 4/29/2019  |           |        | U        | mg/Kg  |         | <9         |               |                               |
| <250um | WG470762LCSS  | LCSS  | Soil Standard | 4/25/2019  | 598       |        | 200.5    | mg/Kg  | 34      | 179-239    | Outside range |                               |
| <250um | WG470843LCSS  | LCSS  | Soil Standard | 4/25/2019  | 8360      |        | 8412     | mg/Kg  | 101     | 4160-12600 |               |                               |
| <250um | WG470843LCSSD | LCSSD | Soil Standard | 4/25/2019  | 8360      |        | 7813     | mg/Kg  | 93      | 4160-12600 |               |                               |
| <250um | WG470843LCSS  | LCSS  | Soil Standard | 4/29/2019  | 8360      |        | 8853     | mg/Kg  | 106     | 4160-12600 |               |                               |
| <250um | WG470843LCSSD | LCSSD | Standard      | 4/29/2019  | 8360      |        | 8284     | mg/Kg  | 99      | 4160-12600 |               |                               |
| <250um | WG471030ICV   | ICV   | Standard      | 4/25/2019  | 0.1       |        | 0.0957   | mg/L   | 96      | 90-110%    |               |                               |
| <250um | WG471030PQV   | PQV   | Standard      | 4/25/2019  | 0.0150195 |        | 0.0137   | mg/L   | 91      | 70-130%    |               |                               |
| <250um | WG471030ICSAB | ICSAB | Standard      | 4/25/2019  | 50.170026 |        | 47.3803  | mg/L   | 94      | 80-120%    |               |                               |
| <250um | WG471030CCV1  | CCV   | Standard      | 4/25/2019  | 0.50065   |        | 0.4655   | mg/L   | 93      | 90-110%    |               |                               |
| <250um | WG471030CCV2  | CCV   | Standard      | 4/25/2019  | 0.50065   |        | 0.4677   | mg/L   | 93      | 90-110%    |               |                               |
| <250um | WG471030CCV3  | CCV   | Standard      | 4/25/2019  | 0.50065   |        | 0.4749   | mg/L   | 95      | 90-110%    |               |                               |
| <250um | WG471219ICV   | ICV   | Standard      | 4/29/2019  | 0.1       |        | 0.0942   | mg/L   | 94      | 90-110%    |               |                               |
| <250um | WG471219PQV   | PQV   | Standard      | 4/29/2019  | 0.0150195 |        | 0.0134   | mg/L   | 89      | 70-130%    |               |                               |
| <250um | WG471219ICSAB | ICSAB | Standard      | 4/29/2019  | 50.170026 |        | 42.4878  | mg/L   | 85      | 80-120%    |               |                               |
| <250um | WG471219CCV1  | CCV   | Standard      | 4/29/2019  | 0.50065   |        | 0.4886   | mg/L   | 98      | 90-110%    |               |                               |
| <250um | WG471219CCV2  | CCV   | Standard      | 4/29/2019  | 0.50065   |        | 0.4903   | mg/L   | 98      | 90-110%    |               |                               |

<sup>a</sup> Alternate calculation to address failure to meet 4x criterion

# Table 3: Barium IVBA results for 2018 Red Dog samples

|                                  |               |                      |   |                               |   |   |  |   | Bioaccess                         |  |                                   |
|----------------------------------|---------------|----------------------|---|-------------------------------|---|---|--|---|-----------------------------------|--|-----------------------------------|
| Sample Identifier                | Grain<br>Size | Solid:Fluid<br>Ratio | Total<br>Conc. In<br>Soil <sup>a</sup><br>(mg/kg) | Mass of<br>Soil Tested<br>(g) | Analyte in<br>Soil<br>Extracted<br>(mg) | Conc. of<br>Analyte in<br>Extract<br>(mg/L) | Mass of<br>Analyte in<br>Extract<br>(mg) | Total Conc. in<br>Soil by XRF <sup>b</sup><br>(mg/kg) | IVBA using<br>Total by XRF<br>(%) | IVBA using<br>Total by<br>3050b<br>(%) | -<br>Ratio of IVBA<br><2mm/<250μm |
| <2mm Particle Size Fraction      |               |                      |   |                               |   |   |  |   |                                   |  |                                   |
| TS-REF-3-COMPOSITE               | <2 mm         | 1:100                | 320   | 0.999                         | 0.32                                    | 1.78  | 0.18                                     | 700   | 30                                | 56                                     | 0.9                               |
| TS-REF-7/TS-REF-11 COMPOSITE     | <2 mm         | 1:100                | 302   | 1.001                         | 0.30                                    | 2.59  | 0.26                                     |   |                                   | 86                                     | 1.0                               |
| TT3-0010 COMPOSITE               | <2 mm         | 1:100                | 2250  | 1.002                         | 2.25                                    | 6.50  | 0.65                                     | 3000  | 22                                | 29                                     | 1.2                               |
| TT3-0100 COMPOSITE               | <2 mm         | 1:100                | 883   | 1.000                         | 0.88                                    | 4.39  | 0.44                                     |   |                                   | 50                                     | 1.0                               |
| TT3-1000 COMPOSITE               | <2 mm         | 1:100                | 251   | 1.000                         | 0.25                                    | 2.26  | 0.23                                     |   |                                   | 90                                     | 1.0                               |
| TT5-0010 COMPOSITE               | <2 mm         | 1:100                | 2140  | 1.003                         | 2.15                                    | 3.49  | 0.35                                     | 2900  | 12                                | 16                                     | 1.0                               |
| TT5-2000 COMPOSITE               | <2 mm         | 1:100                | 154   | 0.997                         | 0.15                                    | 1.37  | 0.14                                     |   |                                   | 89                                     | 1.0                               |
| TT7-0010 COMPOSITE               | <2 mm         | 1:100                | 3510  | 1.002                         | 3.52                                    | 7.01  | 0.70                                     |   |                                   | 20                                     | 1.3                               |
| TT8-0010 COMPOSITE               | <2 mm         | 1:100                | 1590  | 1.000                         | 1.59                                    | 3.72  | 0.37                                     | 2100  | 18                                | 23                                     | 1.1                               |
| TT8-0100 COMPOSITE               | <2 mm         | 1:100                | 992   | 1.003                         | 0.99                                    | 3.99  | 0.40                                     | 2100  | 19                                | 40                                     | 1.1                               |
| TT8-0800/TT8-0900-TT8-1000 COM   | <2 mm         | 1:100                | 203   | 0.999                         | 0.20                                    | 1.79  | 0.18                                     |   |                                   | 88                                     | 1.0                               |
| 18-289 (Y Road Surface)          | <2 mm         | 1:100                | 5700  | 1.003                         | 5.72                                    | 10.5  | 1.05                                     | 21400   | 5                                 | 18                                     | 1.3                               |
| 18-290 (R-Boundary Road Surface) | <2 mm         | 1:100                | 1010  | 0.999                         | 1.01                                    | 2.96  | 0.30                                     | 800   | 37                                | 29                                     | 0.8                               |
| 18-291 (MS-2 Road Surface )      | <2 mm         | 1:100                | 452   | 1.000                         | 0.45                                    | 1.97  | 0.20                                     | 1200  | 16                                | 44                                     | 1.7                               |
| 18-292 (Zinc concentrate)        | <2 mm         | 1:100                | 105   | 1.000                         | 0.11                                    | 1.45  | 0.15                                     | 7700  | 2                                 | 138                                    | 0.5                               |
| 18-293 (Lead concentrate)        | <2 mm         | 1:100                | 185   | 1.002                         | 0.19                                    | 1.5   | 0.15                                     | 8900  | 2                                 | 81                                     | 0.9                               |
| (250m Dertiele Size Freetien     |               |                      |   |                               |   |   |  |   |                                   |  |                                   |
| <250µm Particle Size Fraction    | <050 um       | 1.100                | 200   | 1 000                         | 0.00                                    | 4 74  | 0.47                                     | 600   | 20                                | 50                                     |                                   |
|                                  | <250 µm       | 1:100                | 289   | 1.002                         | 0.29                                    | 1.71  | 0.17                                     | 600   | 30                                | 29                                     |                                   |
| TT2 0040 COMPOSITE               | <250 µm       | 1:100                | 301   | 0.999                         | 0.30                                    | 2.05  | 0.27                                     |   |                                   | 00                                     |                                   |
| TT3-0010 COMPOSITE               | <250 µm       | 1:100                | 2070  | 1.001                         | 2.07                                    | 0.57  | 0.00                                     | 3200  | 21                                | 20                                     |                                   |
| TT3-0100 COMPOSITE               | <250 µm       | 1:100                | 927   | 0.998                         | 0.93                                    | 4.59  | 0.40                                     |   |                                   | 50                                     |                                   |
| TTE 0010 COMPOSITE               | <250 µm       | 1:100                | 237   | 0.999                         | 0.24                                    | 2.15  | 0.22                                     | 2000  |                                   | 91                                     |                                   |
|                                  | <250 µm       | 1.100                | 2020  | 0.999                         | 2.02                                    | 4.00  | 0.41                                     | 3000  | 14                                | 10                                     |                                   |
|                                  | <250 µm       | 1:100                | 103   | 1.000                         | 0.15                                    | 1.41  | 0.14                                     |   |                                   | 92                                     |                                   |
|                                  | <250 µm       | 1:100                | 4470  | 1.003                         | 4.48                                    | 7.14  | 0.71                                     | 0300  | 11                                | 10                                     |                                   |
| TTR 0100 COMPOSITE               | <250 µm       | 1:100                | 1/60  | 1.004                         | 1.77                                    | 3.71  | 0.37                                     | 2200  | 17                                | 21                                     |                                   |
|                                  | <250 µm       | 1:100                | 1060  | 1.000                         | 1.06                                    | 3.97  | 0.40                                     | 1900  | 21                                | 37                                     |                                   |
| 10-0000/118-0900-118-1000 COM    | <250 µm       | 1:100                | 223   | 0.997                         | 0.22                                    | 1.98  | 0.20                                     |   |                                   | 89                                     |                                   |
| 18-289 (Y Road Surface)          | <250 µm       | 1:100                | 1220  | 0.998                         | 0.10                                    | 8.82  | 0.88                                     | 41000   | 2                                 | 14                                     |                                   |
| 10-290 (R-Boundary Road Surface) | <250 µm       | 1:100                | 1330  | 0.998                         | 1.33                                    | 5.01  | 0.50                                     | 2100  | 24                                | 38                                     |                                   |
|                                  | <250 µm       | 1:100                | 1330  | 0.998                         | 1.33                                    | 3.5   | 0.35                                     | 1800  | 19                                | 20                                     |                                   |
| 18-292 (ZINC CONCENTRATE)        | <250 µm       | 1:100                | 04.Z  | 1.001                         | 0.06                                    | 19  | 0.19                                     | 8200<br>0590  | 2                                 | 290                                    |                                   |
| 10-293 (Lead Concentrate)        | <∠o∪ µm       | 1:100                | 148   | 1.001                         | 0.15                                    | 1.34  | 0.13                                     | 9280  | I                                 | 90                                     |                                   |

Note:

<sup>a</sup> Total concentration by 3050b analysis conducted by ACZ Laboratories
 <sup>b</sup> IVBA result calculated using total metals result by x-ray fluorescence at The Mineral Lab.
 --: Not Reported

## Table 4: Barium QA sample results from in vitro barium bioaccessibility testing of 2018 soil samples

| Grain          |               |       |                             | Extraction | QC       | Sample | Measured |       | Percent<br>Recovery | Acceptable |               | Percent<br>Recovery Recalc |
|----------------|---------------|-------|-----------------------------|------------|----------|--------|----------|-------|---------------------|------------|---------------|----------------------------|
| Size           | ACZ ID        | Туре  | Description                 | Date       | Conc.    | Conc.  | Conc.    | Units | (%)                 | Range      | Comments      | (%) <sup>a</sup>           |
| <b>IVBA EX</b> | TRACTION      |       |                             |            |          |        |          |       |                     |            |               |                            |
| <2mm           | WG470546ICB   | ICB   | Blank                       | 4/18/2019  |          |        | U        | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470546CCB1  | CCB   | Blank                       | 4/18/2019  |          |        | U        | mg/L  |                     | < 0.0015   |               |                            |
| <2mm           | WG470546CCB2  | CCB   | Blank                       | 4/18/2019  |          |        | U        | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470546CCB3  | CCB   | Blank                       | 4/18/2019  |          |        | U        | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470956ICB   | ICB   | Blank                       | 4/24/2019  |          |        | U        | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470956CCB1  | CCB   | Blank                       | 4/24/2019  |          |        | U        | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470956CCB2  | CCB   | Blank                       | 4/24/2019  |          |        | U        | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470417LFB   | LFB   | Spiked IVBA Fluid           | 4/18/2019  | 1.002    |        | 0.90272  | mg/L  | 90                  | 80-120%    |               |                            |
| <2mm           | WG470417LFB   | LFB   | Spiked IVBA Fluid           | 4/24/2019  | 1.002    |        | 0.9937   | mg/L  | 99                  | 80-120%    |               |                            |
| <2mm           | L51008-03MS   | MS    | Spiked Soil (L5-1008-03)    | 4/18/2019  | 1.002    | 5.86   | 6.89991  | mg/L  | 104                 | 75-125%    |               | 101                        |
| <2mm           | L51008-03MS   | MS    | Spiked Soil (L5-1008-03)    | 4/24/2019  | 1.002    | 6.5    | 7.457    | mg/L  | 96                  | 75-125%    |               | 99                         |
| <2mm           | L51008-13MS   | MS    | Spiked Soil (L5-1008-13)    | 4/18/2019  | 1.002    | 2.96   | 4.48979  | mg/L  | 153                 | 75-125%    | Outside range | 113                        |
| <2mm           | L51008-13MS   | MS    | Spiked Soil (L5-1008-13)    | 4/24/2019  | 1.002    | 3.22   | 4.8453   | mg/L  | 162                 | 75-125%    | Outside range | 115                        |
| <2mm           | L51008-01DUP  | DUP   | Sample Duplicate            | 4/18/2019  |          | 1.78   | 1.83013  | mg/L  | 103                 | 80-120%    |               |                            |
| <2mm           | L51008-12DUP  | DUP   | Sample Duplicate            | 4/18/2019  |          | 9.29   | 9.18908  | mg/L  | 99                  | 80-120%    |               |                            |
| <2mm           | L51008-01DUP  | DUP   | Sample Duplicate            | 4/24/2019  |          | 1.94   | 1.935    | mg/L  | 100                 | 80-120%    |               |                            |
| <2mm           | L51008-12DUP  | DUP   | Sample Duplicate            | 4/24/2019  |          | 10.5   | 10.289   | mg/L  | 98                  | 80-120%    |               |                            |
| <2mm           | L51008-06SDL  | SDL   | Serial Dilution             | 4/18/2019  |          | 3.49   | 3.5896   | mg/L  | 103                 | 90-110%    |               |                            |
| <2mm           | L51008-08SDL  | SDL   | Serial Dilution             | 4/24/2019  |          | 7.01   | 8.125    | mg/L  | 116                 | 90-110%    |               |                            |
| <2mm           | WG470417PBS   | PBS   | Soil blank                  | 4/18/2019  |          |        | 0.00072  | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470417PBS   | PBS   | Soil blank                  | 4/24/2019  |          |        | U        | mg/L  |                     | <0.009     |               |                            |
| <2mm           | WG470546ICV   | ICV   | Standard                    | 4/18/2019  | 0.05     |        | 0.05262  | mg/L  | 105                 | 90-110%    |               |                            |
| <2mm           | WG470546PQV   | PQV   | Standard                    | 4/18/2019  | 0.002505 |        | 0.00224  | mg/L  | 89                  | 70-130%    |               |                            |
| <2mm           | WG470546ICSA  | ICSA  | Standard                    | 4/18/2019  |          |        | U        | mg/L  |                     | <0.003     |               |                            |
| <2mm           | WG470546ICSAB | ICSAB | Standard                    | 4/18/2019  | 0.02004  |        | 0.01978  | mg/L  | 99                  | 80-120%    |               |                            |
| <2mm           | WG470546CCV1  | CCV   | Standard                    | 4/18/2019  | 0.2505   |        | 0.24197  | mg/L  | 97                  | 90-110%    |               |                            |
| <2mm           | WG470546CCV2  | CCV   | Standard                    | 4/18/2019  | 0.2505   |        | 0.23873  | mg/L  | 95                  | 90-110%    |               |                            |
| <2mm           | WG470546CCV3  | CCV   | Standard                    | 4/18/2019  | 0.2505   |        | 0.23069  | mg/L  | 92                  | 90-110%    |               |                            |
| <2mm           | WG470956ICV   | ICV   | Standard                    | 4/24/2019  | 0.05     |        | 0.0506   | mg/L  | 101                 | 90-110%    |               |                            |
| <2mm           | WG470956PQV   | PQV   | Standard                    | 4/24/2019  | 0.002505 |        | 0.00234  | mg/L  | 93                  | 70-130%    |               |                            |
| <2mm           | WG470956ICSA  | ICSA  | Standard                    | 4/24/2019  |          |        | U        | mg/L  |                     | < 0.003    |               |                            |
| <2mm           | WG470956ICSAB | ICSAB | Standard                    | 4/24/2019  | 0.02004  |        | 0.02331  | mg/L  | 116                 | 80-120%    |               |                            |
| <2mm           | WG470956CCV1  | CCV   | Standard                    | 4/24/2019  | 0.2505   |        | 0.25074  | mg/L  | 100                 | 90-110%    |               |                            |
| <2mm           | WG470956CCV2  | CCV   | Standard                    | 4/24/2019  | 0.2505   |        | 0.25145  | mg/L  | 100                 | 90-110%    |               |                            |
| Total Ba       | rium (3050)   |       |                             |            |          |        |          |       |                     |            |               |                            |
| <2mm           | WG470618ICB   | ICB   | Blank                       | 4/19/2019  |          |        | U        | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470618CCB1  | CCB   | Blank                       | 4/19/2019  |          |        | U        | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470618CCB2  | CCB   | Blank                       | 4/19/2019  |          |        | 0.00088  | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470618CCB3  | CCB   | Blank                       | 4/19/2019  |          |        | 0.00102  | mg/L  |                     | <0.0015    |               |                            |
| <2mm           | WG470959ICB   | ICB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |                     | < 0.0015   |               |                            |
| <2mm           | WG470959CCB1  | CCB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |                     | < 0.0015   |               |                            |
| <2mm           | WG470959CCB2  | CCB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |                     | < 0.0015   |               |                            |
| <2mm           | WG470959CCB3  | CCB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |                     | < 0.0015   |               |                            |
| <2mm           | L51008-16MS   | MS    | Spiked Soil (L51008-16)     | 4/19/2019  | 25.05    | 185    | 173.55   | mg/Kg | -46                 | 75-125%    | Outside range | 83                         |
| <2mm           | L51008-16MSD  | MSD   | Spiked Soil (L51008-16) Dup | 4/19/2019  | 25.05    | 185    | 153.87   | mg/Kg | -124                | 75-125%    | Outside range | 73                         |

## Table 4: Barium QA sample results from in vitro barium bioaccessibility testing of 2018 soil samples

|         |               |       |                             |            |          |        |          |       | Percent  |            |               | Percent          |
|---------|---------------|-------|-----------------------------|------------|----------|--------|----------|-------|----------|------------|---------------|------------------|
| Grain   |               |       |                             | Extraction | QC       | Sample | Measured |       | Recovery | Acceptable |               | Recovery Recalc  |
| Size    | ACZ ID        | Туре  | Description                 | Date       | Conc.    | Conc.  | Conc.    | Units | (%)      | Range      | Comments      | (%) <sup>a</sup> |
| <2mm    | L51008-16MS   | MS    | Spiked Soil (L51008-16)     | 4/25/2019  | 5.01     | 100    | U        | mg/Kg | 0        | 75-125%    | Outside range |                  |
| <2mm    | L51008-16MSD  | MSD   | Spiked Soil (L51008-16) Dup | 4/25/2019  | 5.01     | 100    | U        | mg/Kg | 0        | 75-125%    | Outside range |                  |
| <2mm    | L51008-04SDL  | SDL   | Serial Dilution             | 4/19/2019  |          | 911    | 877.75   | mg/Kg | 96       | 90-110%    | -             |                  |
| <2mm    | L51008-03SDL  | SDL   | Serial Dilution             | 4/25/2019  |          | 2250   | 2283.7   | mg/Kg | 101      | 90-110%    |               |                  |
| <2mm    | WG470471PBS   | PBS   | Soil blank                  | 4/19/2019  |          |        | U        | mg/Kg |          | <0.9       |               |                  |
| <2mm    | WG470471PBS   | PBS   | Soil blank                  | 4/25/2019  |          |        | U        | mg/Kg |          | <0.9       |               |                  |
| <2mm    | WG470471LCSS  | LCSS  | Soil Standard               | 4/19/2019  | 270      |        | 299.6    | mg/Kg | 111      | 223-318    |               |                  |
| <2mm    | WG470471LCSSD | LCSSD | Soil Standard               | 4/19/2019  | 270      |        | 292.5    | mg/Kg | 108      | 223-318    |               |                  |
| <2mm    | WG470471LCSS  | LCSS  | Soil Standard               | 4/25/2019  | 270      |        | 267.9    | mg/Kg | 99       | 223-318    |               |                  |
| <2mm    | WG470471LCSSD | LCSSD | Soil Standard               | 4/25/2019  | 270      |        | 257.2    | mg/Kg | 95       | 223-318    |               |                  |
| <2mm    | WG470618ICV   | ICV   | Standard                    | 4/19/2019  | 0.05     |        | 0.05473  | mg/L  | 109      | 90-110%    |               |                  |
| <2mm    | WG470618PQV   | PQV   | Standard                    | 4/19/2019  | 0.002505 |        | 0.00251  | mg/L  | 100      | 70-130%    |               |                  |
| <2mm    | WG470618ICSA  | ICSA  | Standard                    | 4/19/2019  |          |        | U        | mg/L  |          | <0.003     |               |                  |
| <2mm    | WG470618ICSAB | ICSAB | Standard                    | 4/19/2019  | 0.02004  |        | 0.02225  | mg/L  | 111      | 80-120%    |               |                  |
| <2mm    | WG470618CCV1  | CCV   | Standard                    | 4/19/2019  | 0.2505   |        | 0.24812  | mg/L  | 99       | 90-110%    |               |                  |
| <2mm    | WG470618CCV2  | CCV   | Standard                    | 4/19/2019  | 0.2505   |        | 0.24542  | mg/L  | 98       | 90-110%    |               |                  |
| <2mm    | WG470618CCV3  | CCV   | Standard                    | 4/19/2019  | 0.2505   |        | 0.2504   | mg/L  | 100      | 90-110%    |               |                  |
| <2mm    | WG470959ICV   | ICV   | Standard                    | 4/25/2019  | 0.05     |        | 0.05063  | mg/L  | 101      | 90-110%    |               |                  |
| <2mm    | WG470959PQV   | PQV   | Standard                    | 4/25/2019  | 0.002505 |        | 0.00223  | mg/L  | 89       | 70-130%    |               |                  |
| <2mm    | WG470959ICSA  | ICSA  | Standard                    | 4/25/2019  |          |        | U        | mg/L  |          | < 0.003    |               |                  |
| <2mm    | WG470959ICSAB | ICSAB | Standard                    | 4/25/2019  | 0.02004  |        | 0.01915  | mg/L  | 96       | 80-120%    |               |                  |
| <2mm    | WG470959CCV1  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.24461  | mg/L  | 98       | 90-110%    |               |                  |
| <2mm    | WG470959CCV2  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.24709  | mg/L  | 99       | 90-110%    |               |                  |
| <2mm    | WG470959CCV3  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.24218  | mg/L  | 97       | 90-110%    |               | _                |
| IVBA EX | RACTION       |       |                             |            |          |        |          |       |          |            |               |                  |
| <250um  | WG471024ICB   | ICB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG471024CCB1  | CCB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG471024CCB2  | CCB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG471024CCB3  | CCB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG471024CCB4  | CCB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG470840LFB   | LFB   | Spiked IVBA Fluid           | 4/25/2019  | 1.002    |        | 0.9481   | mg/L  | 95       | 80-120%    |               |                  |
| <250um  | L51009-03MS   | MS    | Spiked Soil (L51009-03)     | 4/25/2019  | 1.002    | 6.57   | 7.3388   | mg/L  | 77       | 75-125%    |               |                  |
| <250um  | L51009-16MS   | MS    | Spiked Soil (L51009-16)     | 4/25/2019  | 1.002    | 1.34   | 1.7829   | mg/L  | 44       | 75-125%    | Outside range | 76               |
| <250um  | L51009-01DUP  | DUP   | Soil Dup                    | 4/25/2019  |          | 1.71   | 1.7064   | mg/L  | 100      | 80-120%    |               |                  |
| <250um  | L51009-15DUP  | DUP   | Soil Dup                    | 4/25/2019  |          | 1.9    | 1.7834   | mg/L  | 94       | 80-120%    |               |                  |
| <250um  | L51009-06SDL  | SDL   | Serial Dilution             | 4/25/2019  |          | 4.06   | 3.907    | mg/L  | 96       | 90-110%    |               |                  |
| <250um  | WG470840PBS   | PBS   | Soil blank                  | 4/25/2019  |          |        | U        | mg/L  |          | <0.009     |               |                  |
| <250um  | WG471024ICV   | ICV   | Standard                    | 4/25/2019  | 0.05     |        | 0.05097  | mg/L  | 102      | 90-110%    |               |                  |
| <250um  | WG471024PQV   | PQV   | Standard                    | 4/25/2019  | 0.002505 |        | 0.00223  | mg/L  | 89       | 70-130%    |               |                  |
| <250um  | WG471024ICSA  | ICSA  | Standard                    | 4/25/2019  |          |        | U        | mg/L  |          | <0.003     |               |                  |
| <250um  | WG471024ICSAB | ICSAB | Standard                    | 4/25/2019  | 0.02004  |        | 0.01855  | mg/L  | 93       | 80-120%    |               |                  |
| <250um  | WG471024CCV1  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.25291  | mg/L  | 101      | 90-110%    |               |                  |
| <250um  | WG471024CCV2  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.25367  | mg/L  | 101      | 90-110%    |               |                  |
| <250um  | WG471024CCV3  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.24974  | mg/L  | 100      | 90-110%    |               |                  |
| <250um  | WG471024CCV4  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.24784  | mg/L  | 99       | 90-110%    |               |                  |

## Table 4: Barium QA sample results from in vitro barium bioaccessibility testing of 2018 soil samples

|         |               |       |                             |            |          |        |          |       | Percent  |            |               | Percent          |
|---------|---------------|-------|-----------------------------|------------|----------|--------|----------|-------|----------|------------|---------------|------------------|
| Grain   |               |       |                             | Extraction | QC       | Sample | Measured |       | Recovery | Acceptable |               | Recovery Recalc  |
| Size    | ACZ ID        | Туре  | Description                 | Date       | Conc.    | Conc.  | Conc.    | Units | (%)      | Range      | Comments      | (%) <sup>a</sup> |
| TOTAL B | ARIUM (3050)  |       |                             |            |          |        |          |       |          |            |               |                  |
| <250um  | WG471030ICB   | ICB   | Blank                       | 4/25/2019  |          |        | U        | ma/L  |          | <0.0015    |               |                  |
| <250um  | WG471030CCB1  | CCB   | Blank                       | 4/25/2019  |          |        | Ŭ        | ma/L  |          | < 0.0015   |               |                  |
| <250um  | WG471030CCB2  | CCB   | Blank                       | 4/25/2019  |          |        | U        | mg/L  |          | < 0.0015   |               |                  |
| <250um  | WG471030CCB3  | CCB   | Blank                       | 4/25/2019  |          |        | 0.00071  | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG471219ICB   | ICB   | Blank                       | 4/29/2019  |          |        | U        | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG471219CCB1  | CCB   | Blank                       | 4/29/2019  |          |        | U        | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG471219CCB2  | CCB   | Blank                       | 4/29/2019  |          |        | U        | mg/L  |          | <0.0015    |               |                  |
| <250um  | WG470762LFB2  | LFB   | Spiked IVBA Fluid           | 4/25/2019  | 0.0501   |        | 0.04564  | mg/Kg | 91       | 80-120%    |               |                  |
| <250um  | WG470762LFBD2 | LFBD  | Spiked IVBA Fluid           | 4/25/2019  | 0.0501   |        | 0.04656  | mg/Kg | 93       | 80-120%    |               |                  |
| <250um  | L51009-09MS   | MS    | Spiked Soil (L51009-09)     | 4/25/2019  | 25.05    | 1760   | 1821.97  | mg/Kg | 247      | 75-125%    | Outside range | 102              |
| <250um  | L51009-09MSD  | MSD   | Spiked Soil (L51009-09) Dup | 4/25/2019  | 25.05    | 1760   | 1797.93  | mg/Kg | 151      | 75-125%    | Outside range | 101              |
| <250um  | L51009-09MS   | MS    | Spiked Soil (L51009-09)     | 4/29/2019  | 25       | 1730   | 1801     | mg/Kg | 284      | 75-125%    | Outside range | 103              |
| <250um  | L51009-09MSD  | MSD   | Spiked Soil (L51009-09) Dup | 4/29/2019  | 25       | 1730   | 1824.2   | mg/Kg | 377      | 75-125%    | Outside range | 104              |
| <250um  | L51009-03SDL  | SDL   | Serial Dilution             | 4/25/2019  |          | 2570   | 2390.5   | mg/Kg | 93       | 90-110%    | -             |                  |
| <250um  | L51009-11SDL  | SDL   | Serial Dilution             | 4/29/2019  |          | 272    | 267.35   | mg/Kg | 98       | 90-110%    |               |                  |
| <250um  | WG470762PBS   | PBS   | Soil blank                  | 4/25/2019  |          |        | U        | mg/Kg |          | <0.9       |               |                  |
| <250um  | WG470843PBS   | PBS   | Soil blank                  | 4/25/2019  |          |        | U        | mg/Kg |          | <0.9       |               |                  |
| <250um  | WG470843PBS   | PBS   | Soil blank                  | 4/29/2019  |          |        | U        | mg/Kg |          | <0.9       |               |                  |
| <250um  | WG470843LCSS  | LCSS  | Soil Standard               | 4/25/2019  | 270      |        | 265.1    | mg/Kg | 98       | 223-318    |               |                  |
| <250um  | WG470843LCSSD | LCSSD | Soil Standard               | 4/25/2019  | 270      |        | 250.1    | mg/Kg | 93       | 223-318    |               |                  |
| <250um  | WG470843LCSS  | LCSS  | Soil Standard               | 4/29/2019  | 270      |        | 295.8    | mg/Kg | 110      | 223-318    |               |                  |
| <250um  | WG470843LCSSD | LCSSD | Soil Standard               | 4/29/2019  | 270      |        | 278.1    | mg/Kg | 103      | 223-318    |               |                  |
| <250um  | WG471030ICV   | ICV   | Standard                    | 4/25/2019  | 0.05     |        | 0.05019  | mg/L  | 100      | 90-110%    |               |                  |
| <250um  | WG471030PQV   | PQV   | Standard                    | 4/25/2019  | 0.002505 |        | 0.00224  | mg/L  | 89       | 70-130%    |               |                  |
| <250um  | WG471030ICSA  | ICSA  | Standard                    | 4/25/2019  |          |        | U        | mg/L  |          | <0.003     |               |                  |
| <250um  | WG471030ICSAB | ICSAB | Standard                    | 4/25/2019  | 0.02004  |        | 0.01982  | mg/L  | 99       | 80-120%    |               |                  |
| <250um  | WG471030CCV1  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.23729  | mg/L  | 95       | 90-110%    |               |                  |
| <250um  | WG471030CCV2  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.2386   | mg/L  | 95       | 90-110%    |               |                  |
| <250um  | WG471030CCV3  | CCV   | Standard                    | 4/25/2019  | 0.2505   |        | 0.23479  | mg/L  | 94       | 90-110%    |               |                  |
| <250um  | WG471219ICV   | ICV   | Standard                    | 4/29/2019  | 0.05     |        | 0.05251  | mg/L  | 105      | 90-110%    |               |                  |
| <250um  | WG471219PQV   | PQV   | Standard                    | 4/29/2019  | 0.002505 |        | 0.00233  | mg/L  | 93       | 70-130%    |               |                  |
| <250um  | WG471219ICSA  | ICSA  | Standard                    | 4/29/2019  | 0.00055  |        | 0.00055  | mg/L  | 100      | < 0.003    |               |                  |
| <250um  | WG471219ICSAB | ICSAB | Standard                    | 4/29/2019  | 0.02004  |        | 0.02068  | mg/L  | 103      | 80-120%    |               |                  |
| <250um  | WG471219CCV1  | CCV   | Standard                    | 4/29/2019  | 0.2505   |        | 0.2452   | mg/L  | 98       | 90-110%    |               |                  |
| <250um  | WG471219CCV2  | CCV   | Standard                    | 4/29/2019  | 0.2505   |        | 0.24592  | mg/L  | 98       | 90-110%    |               |                  |

<sup>a</sup> Alternate calculation to address failure to meet 4x criterion